LUMINESCENCE STUDIES OF ION-IMPLANTED GALLIUM NITRIDE AND ALLUMINUM GALLIUM NITRIDE

THESIS

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AFIT/GAP/ENP/03-02

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Abstract

Recently, research on the wide bandgap semiconductors such as GaN and $Al_xGa_{1-x}N$ became very popular for their applications on various devices. Therefore comprehensive and systematic luminescence studies of Si implanted $Al_xGa_{1-x}N$, Mg doped GaN, and Si+N implanted GaN grown on sapphire substrates by molecular beam epitaxial method have been made as a function of ion dose and anneal temperature. The ions were implanted at 200 keV with doses ranging from $1x10^{13}$ to $1x10^{15}$ cm^{-2} at room temperature. The samples were proximity cap annealed from 1200 to 1350 ^oC with a 500 Å thick AlN cap in a nitrogen environment. It has been found that the optical activation and implantation damage recovery are highly dependent upon ion dose and anneal temperature. The results of luminescence measurements on $Al_xGa_{1-x}N$ made at 5 K by both photoluminescence and cathodoluminescence show that nearly complete implantation damage recovery can only be obtained after annealing at 1350 ^oC. The Si+N implanted GaN showed only a small amount of optical activation in the cathodoluminescence measurements at 1250 to 1350 ^oC. Mg doped GaN results indicated that optical activation of the Mg ions was not prevalent, and ion implantation damage might not have been removed completely at 1350 °C. The results also indicate that current AlN cap protected the implanted $Al_xGa_{1-x}N$ layer very well during high temperature annealing without creating any significant annealing damage. These luminescence observations are consistent with the results of electrical activation studies made on these samples.

LUMINESCENCE STUDIES OF ION-IMPLANTED
GALLIUM NITRIDE AND ALLUMINUM GALLIUM NITRIDE

I. Introduction

Electronic and optical devices are extensively used for many applications in today's world. Their purpose is generally dependent on their design and the material properties of the semiconductors from which they are built. The two most common materials used are silicon and gallium arsenide. Both are used because of their ability to be grown in bulk (thus making them inexpensive) and the fact that they work well at room temperature. However, Si, GaAs, and their alloys cannot function in elevated temperatures or harsh environments. (Mohammed, 1995) Wide bandgap III-V semiconductor materials such as gallium nitride, aluminum nitride, and aluminum gallium nitride have received much attention because of their superior material parameters, i.e. they can be used in the conditions where the typical semiconductor materials cannot. The III-V nitrides are suited for high temperature, high power, and high frequency electronic and optical devices. Of the nitrides, AlGaN and GaN are expected to best meet the needs of future military radar and communications systems. (Fellows, 2000)

AlGaN and GaN materials are of interest because they can be used for short wavelength optoelectronic applications that are beyond the range of present semiconductor devices. The AlGaN and GaN materials have these desirable characteristics: large energy direct bandgap, physical hardness, radiation hardness, large heterojunction offsets, high charge carrier saturation velocity, low dielectric constants,

high thermal conductivity, and high chemical, thermal, and mechanical stability. The bandgap energies of AlGaN range from 3.4 eV (GaN) to 6.2 eV (AlN). (McFall, 2000) Current semiconductor optical devices routinely operate between the infrared and green wavelengths. This range would be increased with the advent of AlGaN and GaN materials to include the blue and ultraviolet wavelengths, thus allowing emitters and detectors to emit and detect one of the primary colors of the visible spectrum. AlGaN and GaN would allow for the fabrication of light emitting diodes (LEDs), diode lasers, and detectors operating at short wavelengths. (Ahoujja, 2002) This would have a great impact on imaging applications. (Strite, 1992) These are essential devices for developing full color displays, high density optical storage technologies, and devices for signal and illumination applications. LEDs are very reliable and are currently used for displays, lighting, indicator lights to traffic lights, accelerated photosynthesis, and the production of white light. (Mohammed, 1995)

Therefore AlGaN and GaN devices are suitable for military applications. Applications range from UV detectors in plume detection and threat warning systems to LEDs, lasers, communication electronics, and field effect transistors. Tanks, ships, aircraft, and military communication devices can all utilize and benefit from AlGaN devices. (McFall, 2000)

However, there are disadvantages to using AlGaN and GaN in semiconductor devices. There is a lack of understanding of the native defects, and there are also doping problems. The object of this research is to determine the optical characteristics of AlGaN and GaN samples implanted with various Si and Si+N doses. The mole fraction of Al in AlGaN range from x = 0.1 to x = 0.3. Primarily cathodoluminescence (CL) as well as

photoluminescence (PL) measurements will be used to determine the optical properties of the material. CL and PL provide a means of excitation of electron-hole pairs in the material. The resulting radiative recombination of the electrons and holes is dependent on the availability of the transitional energy levels. The presence/absence of specific energy levels indicates the presence/absence of the impurity species.

In order to effectively use the technology made possible by AlGaN and GaN, the optical properties of undoped and doped material must be fully understood. This research has performed a comprehensive and systematic study of the optical activation of $Al_xGa_{1-x}N$ (x = 0.1 to 0.3) and GaN as a function of ion species, ion implantation, and annealing temperature. Results of this study will allow a greater understanding and ability to use GaN and $Al_xGa_{1-x}N$ in enhanced optical device applications.

II. Background

This chapter provides a brief background in the theory of semiconductors relating to GaN and AlGaN, and the methods used to characterize them.

Crystal Structure

Semiconductor atoms arrange themselves in three dimensional crystalline structures. There are fourteen unique structures called Bravais lattices. Within these Bravais lattices there are seven distinct symmetry groups. (McKelvey, 1993) GaN and AlGaN generally have a Wurtzite structure comprising of two hexagonal Bravais lattices intertwined. Each lattice has a unit cell, or region of the crystal that is reproducible throughout the crystal. The lattice constant is the length of a unit cell in the lattice. Closely matching lattice constants during growth is important when one semiconductor is epitaxially grown on another. (McKelvey, 1993)

Crystal Growth

Most bulk semiconductor materials are grown under strict thermodynamic equilibrium conditions. Bulk crystals are then cut into thin wafers and their surfaces are polished. Semiconductor devices are fabricated on epitaxially grown material on the bulk semiconductors or substrates. (Fellows, 2001)

The particular growth method used for the samples on this experiment was molecular beam epitaxy (MBE). This growth technique is used to provide precise control over the chemical composition and doping level. A thin single layer of crystal is grown on top of the buffer layer that is grown on the substrate. This technique is preferred for III-V nitride compounds because bulk nitrides are very difficult to grow. (McFall, 2000) Undoped AlGaN is grown on a two inch diameter sapphire substrate by MBE using an rf atomic nitrogen plasma source by SVT Associates. (Fellows, 2002) Because of the reactivity of Al, a pure source material and an oxygen free environment are necessary to grow AlGaN. The mole fraction of Al can be varied, thus rising to the common notation $Al_xGa_{1-x}N$. The high mole fraction is required for operation into the UV. GaN will also be grown in the same manner as AlGaN.

Ion Implantation

Dopability of GaN and AlGaN has not been thoroughly researched to date. There have been difficulties in successfully doping p-type GaN and AlGaN, but doping is possible for n-type GaN and AlGaN up to $x = 0.5$. There are three methods to dope a material: epitaxial growth, diffusion, and ion implantation. One of the doping methods of $Al_xGa_{1-x}N$ and GaN is by ion implantation. The advantages of ion implantation include accurate doping level control, excellent uniformity, and the ability to dope shallow layers with very high impurity concentrations. However, ion implantation does have its disadvantages. Radiation damage occurs to the crystal structure during implantation, thus altering the electrical and optical properties of the semiconductor. Also, implanted layers

are generally limited to the surface. Greater penetration depths can be achieved at the expense of increased radiation damage, which cause defects in the crystal. As the ion travels in the semiconductor, it collides and leaves behind a trail of displaced host atoms. Simple or point defects affect only one host atom. They include vacancies (V_{Ga}, V_{Al} or V_N), interstitials, or antisites. Complex defects occur when two or more point defects combine. These defects can destroy the efficiency of optical and electrical devices by offering preferential non-radiative recombination routes. (Fellows, 2000)

In order to make the implanted ions electrically active and to remove the radiation damage, the samples must be annealed. This procedure repairs the radiation damage of the ion implantation so that the optical properties of the doped GaN and $Al_xGa_{1-x}N$ can be observed. (Fellows, 2001)

Bandgap Energy

The conductivity of semiconductors is dependent upon the occupancy of the energy levels or bands. The highest occupied band is called the valence band, and the lowest unoccupied band is the conduction band. The energy difference between the two bands is the bandgap energy. For semiconductors, this bandgap energy can range from a few meV to almost 6 eV. Because of the wide range of energies, semiconductors are further classified into narrow, medium, and wide bandgap energies. Wide bandgap semiconductors are those with bandgap energies greater than 2.2 eV, therefore making excellent sources for blue/ultraviolet LED's, lasers, and detectors. (Fellows, 2001) There are sub-band energies located between the valence and conduction bands, allowing

for many other electron and hole recombinations. Yoshida *et al.* observed the $Al_xGa_{1-x}N$ energy bandgap to deviate upward as the mole fraction of Al was increased. Hagen *et al.* and Koide *et al.* observed that as the mole fraction of Al was increased, the energy bandgap varied by

$$E_g(x) = xE_g(AlN) + (1-x)E_g(GaN) - bx(1-x) \quad (1)$$

where $E_g(GaN) = 3.39$ eV, $E_g(AlN) = 6.20$ eV, and $b = 1.0 \pm 0.3$ eV. Lowercase b is the bowing parameter, and x is the mole fraction of Al. (Mohammed, 1995)

Luminescence

Luminescence is the simplest way to determine what defects and impurity levels exist. Two types were used in this research, photoluminescence (PL) and cathodoluminescence (CL). Both monitor the radiative recombination of electrons and holes in semiconductors, providing information on the impurity states in the energy gap. (Keefer, 1987) The two methods are distinguished by their excitation source. In photoluminescence the excitation source is a laser whose energy is generally 0.1 –1 eV greater than the energy bandgap. In this study a 275 nm Ar ion laser line was used to excite GaN and AlGaN with mole fractions less than 0.3. A typical experimental set-up of PL is shown in figure 2-1. (Fellows, 2001)

The excitation source for cathodoluminescence is an electron beam which can be thousands of eVs greater than the energy bandgap. Because of this, CL can be used when the energy bandgap is too wide for a laser ($x > 0.3$) and also when it is desired the beam

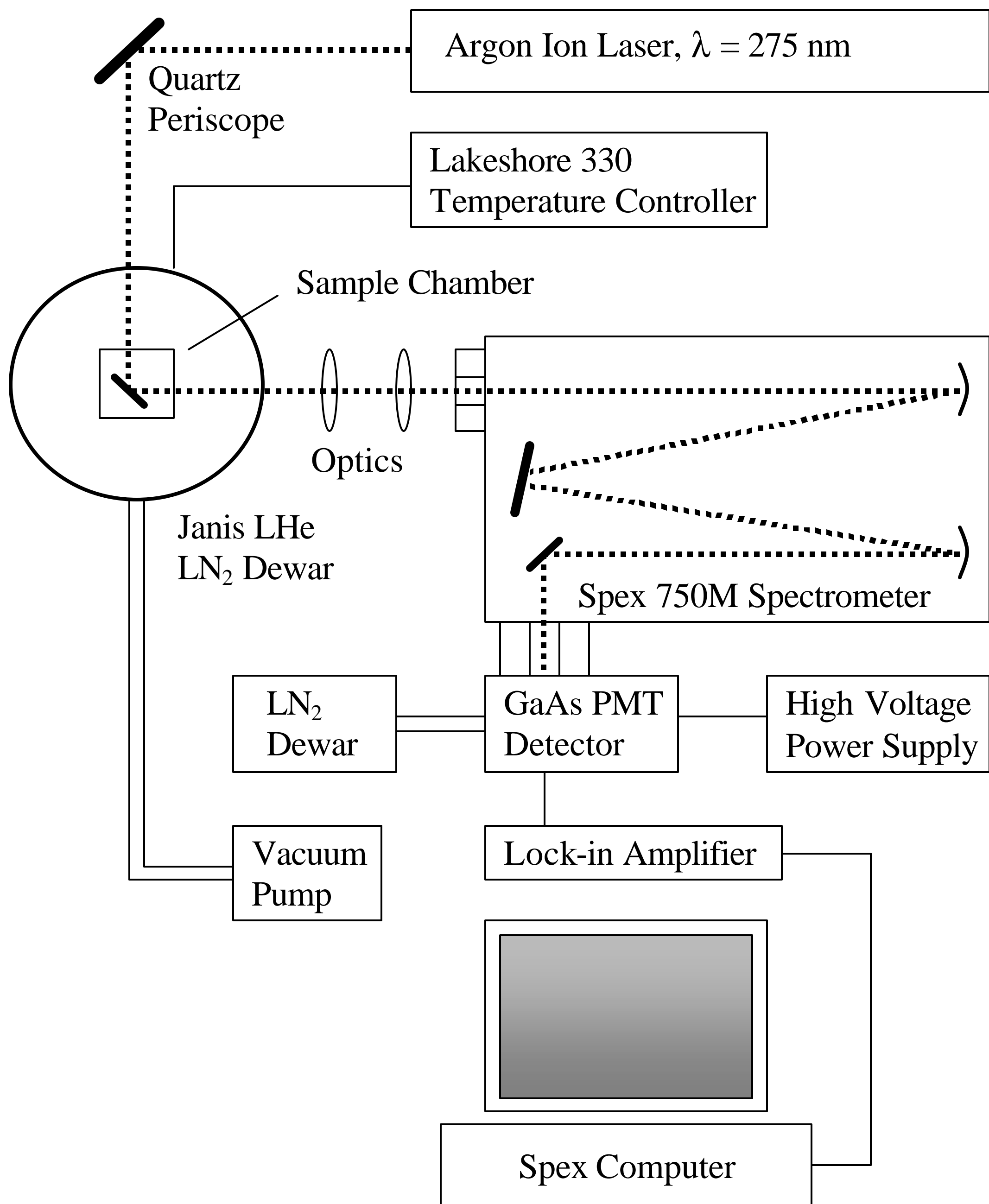

Figure 2-1. Photoluminescence experimental set-up (Fellows, 2001)

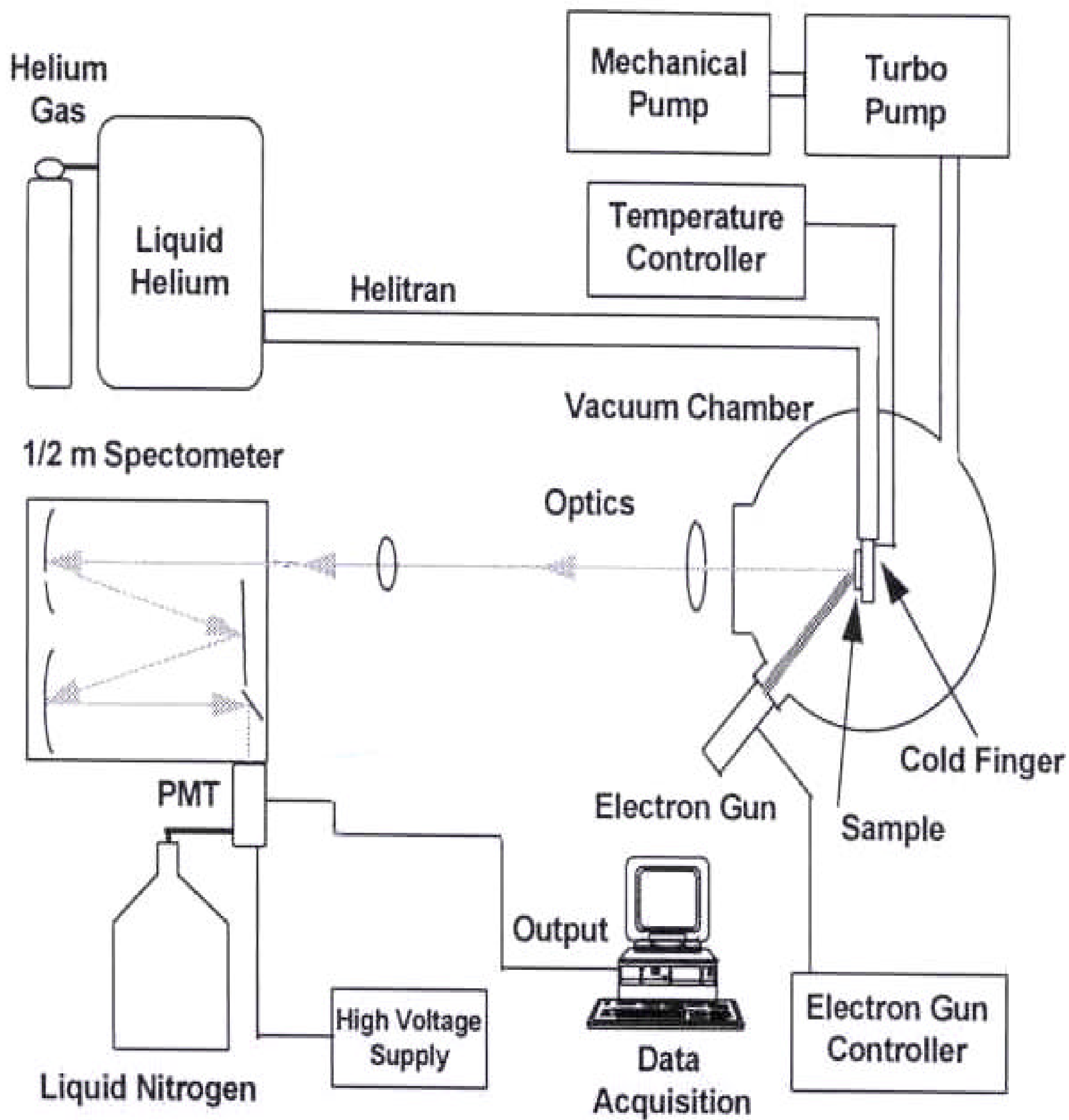

Figure 2-2. Cathodoluminescence experimental set-up (McFall, 2000)

penetrate the material more deeply than the laser. A typical CL set-up is shown in figure 2-2. (Fellows, 2001)

Radiation Transitions

Electron and hole recombinations can emit photons at several different energies because of the many energy levels in the crystal. The possible radiative transitions are shown and labeled in figure 2-3. (McFall, 2000)

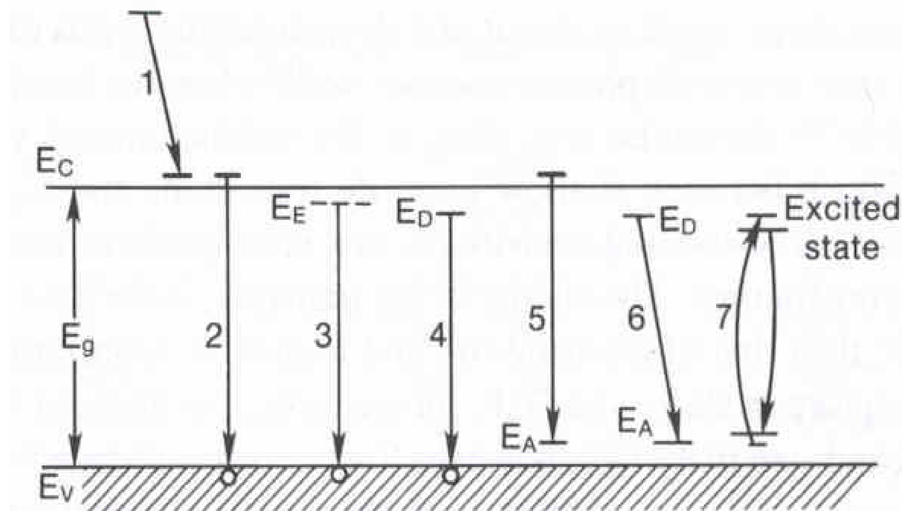

Figure 2-3. Radiation Transitions diagram for all possible energy levels between the conduction and valence band

Transition 1 occurs when an electron excited above the conduction band-edge drops to the bottom of the conduction band (E_c). A phonon is usually emitted in this process.

Transition 2 is an interband transition. An electron in the conduction band (E_c) recombines with a hole in the valence band (E_v). The photon emitted has approximately the same energy as the bandgap energy (E_g). Transition 2 is not a dominant transition because the emitted photons can be reabsorbed by the crystal.

Transition 3 is an exciton decay. An exciton is an electron-hole pair where the electron orbits the hole. Sharp peaks in the absorption edge of direct bandgap semiconductors indicate excitons, which are produced by photon absorption. Excitons are generally observed at low (liquid He) temperatures. Above a certain temperature, excitons ionize and the electron-hole pair splits. There are two types of excitons, free and bound. Free excitons are electron-hole pairs that move through the crystal with a kinetic energy. The free exciton recombination energy can be calculated from the equation,

$$h\nu = E_g - E_{ex}, \tag{2}$$

where h? is the photon energy, E_g is the bandgap energy, and E_{ex} is the free exciton binding energy. The binding energy of an exciton is less than the binding energies of acceptors or donors, because the reduced mass of an exciton is less than the effective mass of the electron. When a free hole is attaached to a neutral donor, a bound exciton is formed. The donor electron binds to the free hole and both orbit the donor ion. A bound exciton can also occur when a free electron is attached to a neutral acceptor. The bound exciton transition energy is given by,

$$h\nu = E_g - E_{ex} - E_b \, , \quad (3)$$

where h? is the photon energy, E_g is the bandgap energy, E_{ex} is the free exciton binding energy, and E_b is the bound exciton binding energy. Bound excitons do not have any kinetic energy and therefore their transitions produce narrower peaks than free exc iton transitions.

Transitions 4 and 5 are impurity transitions. Impurity transitions can start and/or end on donor and acceptor levels in the bandgap. Transition 4 is an electron falling from the donor level to the valence band, and transition 5 is an electron falling from the conduction band to the acceptor level. These transitions are free to bound recombinations. If the impurity level is close to the conduction or valence band, called a shallow donor or acceptor, it can be hard to distinguish between the impurity and the band edge. If there is a high concentration of impurities, the impurity levels assimilate into the closest intrinsic band, making it difficult to determine the origin of the transition, which can be direct or indirect. A transitio n in a direct bandgap semiconductor occurs where the wave vector k is the same for the maximum of the valence band and the minimum of the conduction band. The photon energy for the direct transition is

$$h\nu = E_g - E_i \, , \quad (4)$$

where h? is the photon energy, E_g is the bandgap energy, and E_i is the ionization energy of the acceptor or donor. An indirect transition occurs when the wave vector k does not have the same value for the band extrema. A phonon is required to conserve momentum. The photon energy for this transition is given by,

$$h\nu = E_g - E_i - E_p \, , \quad (5)$$

where h? is the photon energy, E_g is the bandgap energy, E_i is the ionization energy of the acceptor or donor, and E_p is the emitted phonon energy.

The donor-acceptor pair transition is number 6. An electron from a neutral donor recombines with a hole on a neutral acceptor. The separation between the acceptor and donor also effects the energy of the photon and therefore the energy of the electron-hole pair. To determine this energy the equation,

$$h\nu = E_g - (E_a + E_d) + \frac{e^2}{\epsilon * r} \tag{6}$$

is used where h? is the photon energy, E_g is the bandgap energy, E_a is the acceptor level energy, E_d is the donor level energy, e is the charge of the electron, e is the dielectric constant, and r is the separation distance between the donor and acceptor. (McFall, 2000)

III. Experimental Procedures

This chapter describes the experimental procedures from material growth to ion implantation, annealing, ohmic contacts and finally photoluminescence and cathodoluminescence measurements. In this study, both GaN and $Al_xGa_{1-x}N$ (x = 0.1, 0.2, 0.3) were studied. Both were implanted with various doses of magnesium, silicon and/or nitrogen, and annealed at various temperatures.

Sample Preparation for Ion Implantation

Samples of undoped GaN and $Al_xGa_{1-x}N$ were grown by SVT Associates on two inch sapphire substrates via MBE using an rf atomic nitrogen plasma source. Approximately two microns of epilayer were grown on each substrate. A 500 Å thick AlN cap was then grown on the sample in a nitrogen rich environment. In the cleanroom, the Ti coating on the backside of the substrate was removed using HF after the samples had preliminary CL measurements to insure proper quality of the materials. The samples were cleaned using acetone, methanol, and blown dry with N_2. Photoresist 1812 was spun onto the samples to help protect against debris during cutting. After applying the photoresist, the samples were baked at 100º C for five minutes. The samples were then cut into quarters using a diamond wire saw. The residue remaining on the samples after cutting was removed by soaking it in an acetone:distilled water (1:3) bath and then an ultrasonic cleaning for one minute. The samples were then cleaned using acetone, methanol, and blown dry with N_2. Ion implantation was performed by Implant Sciences

Corporation. The undoped GaN samples were co-implanted with Si and N at three doses. The doses used were 1 x 10^{13} Si / 9 x 10^{12} N, 1 x 10^{14} Si / 9 x 10^{13} N, and 1 x 10^{15} Si / 9 x 10^{14} N ions per cm^2. P-type GaN samples (doped with Mg) were implanted with Mg at doses of 5 x 10^{13}, 5 x 10^{14} , and 5 x 10^{15} ions per cm^2. $Al_xGa_{1-x}N$ samples were implanted with various Si doses at 1 x 10^{13}, 1 x 10^{14}, and 1 x 10^{15} ions per cm^2.

Anneal and Ohmic Contact Procedures

Samples were cut into 5.2 mm x 5.2 mm size using the diamond wire saw, and the same cleaning procedures were used on these square samples to remove debris and residue from the cutting as the quarters. Each square sample was then scribed with a distinguishing mark on the sapphire substrate for identification. Pairs of samples that matched perfectly in size were set aside for annealing. Each pair was wrapped tightly face to face using a 5 mil Ta wire, providing a tight proximity cap. Two Oxy-Gon furnaces were utilized. The first was the furnace provided by the Air Force Research Laboratory (AFRL), Materials and Manufacturing Directorate. The second was the furnace purchased by the Air Force Institute of Technology (AFIT). Samples were annealed from 1200 ºC to 1350 ºC for times between 30 – 120 seconds in a nitrogen rich environment. For both furnaces, samples were placed on a SiC coated graphite pedestal. To remove oxygen from the environment, a vacuum was pulled to 10^{-3} torr in the Oxy-Gon sample chamber, the furnace was backfilled with nitrogen and then a vacuum pulled a second time. The samples were annealed at an over-pressure of 1 psig of N_2. To prevent oxidation of the AlN cap, samples were cooled in the furnace chamber until

reaching room temperature. Detailed sample cutting and cleaning procedures are described in Appendix B, and furnace procedures are listed in Appendix C.

After unwrapping the sample by removing the Ta wire, the AlN cap was selectively etched using a 0.5M KOH solution at 90 ºC. GaN was soaked in the KOH solution for 5 minutes, and $Al_xGa_{1-x}N$ was soaked for 10 minutes. The amount of time to remove the AlN cap was determined using a depth profiler. Five minutes was sufficient to remove the 500 Å AlN cap for GaN at a rate of 100 Å per minute. $Al_xGa_{1-x}N$ had the same rate of 100 Å per minute, but had as much as 500 more angstroms on the cap than requested. Therefore 10 minutes was used to sufficiently remove the cap. The samples were then cleaned and blown dry with N_2 before being placed in an aqua regia solution of $HCl:HNO_3$ (3:1) for two minutes to remove oxides and prepare the sample surface. Samples were then mounted on a van der Pauw contact shadow mask plate for contact evaporation. The equipment used was a BOC Edwards four hearth 306 electron beam evaporator. A pressure of 2×10^{-7} torr was initially reached in the evaporator, then Ni/Au contacts were placed on p-type GaN, Ti/Al contacts placed on n-type GaN, and Ti/Al/Ti/Au contacts were placed on $Al_xGa_{1-x}N$. The contacts were approximately 250 microns in radius. The ohmic contacts were flash annealed in an AG Associates Heatpulse 610 RTA face up on a Si wafer at 700 ºC for 30 seconds for $Al_xGa_{1-x}N$ and 900 ºC for 30 seconds for GaN. Contacts were placed on the four corners of the samples to allow a means of obtaining Hall effect measurements; however, this experiment was primarily concerned with the luminescence measurements of the semiconductor materials. I-V curves were measured on the contacts to determine linearity using a HP

4155A Parameter Analyzer on a probe station. Detailed step by step procedures followed in this experiment can be found in Appendix D.

Photoluminescence Measurements

The excitation source for PL was a Spectra-Physics argon ion laser line at 275 nm. Measurements were conducted at T = 3 K except for temperature dependent measurements, which were taken from T = 3 to 300 K. The PL signal was dispersed with a 750 mm spectrometer using a 5000 Å blazed grating and the signal was detected using a liquid nitrogen cooled GaAs photomultiplier tube (PMT) detector. Luminescence was collected from 1.8 to 4.2 eV (7000 to 2800 Å) using a spectrometer step of 2 Å with spectrometer slits set at 400 μm. Detailed PL experimental procedures are in Appendix E.

Cathodoluminescence Measurements

The excitation source for CL was a Kimball electron gun with an electron beam energy ranging from 2.5 to 10 keV, a beam diameter of 2 mm, and an emission current of 50 μA. Measurements were conducted at T = 5 K. The CL signal was dispersed with a 500 mm spectrometer, and was detected using a liquid nitrogen cooled GaAs PMT detector. The grating was also blazed at 5000 Å. Luminescence data was collected from 1.8 to 4.1 eV (7000 to 3000 Å) for GaN and 1.8 to 4.6 eV (7000 to 2500 Å) for $Al_xGa_{1-x}N$ using a spectrometer step size of 2 Å and a range of slit widths from 30 – 400 μm

depending on the luminescence intensity and the spectral resolution. Detailed CL procedures are listed in Appendix E.

IV. Results

Bandgap Energies

The theoretical bandgap energies versus the experimental bandgap energies for $Al_xGa_{1-x}N$ and GaN are in Table 4-1 and Table 4-2, respectively. The theoretical bandgap energy was determined using equation (1) and Mohhamed, 1995. The experimental bandgap energy was taken at 5 K and determined using the luminescence obtained by CL.

$Al_xGa_{1-x}N$	Theoretical Bandgap Energy	Experimental Bandgap Energy
X = 0.1	3.748 eV	3.745 eV
X = 0.2	4.021 eV	3.995 eV
X = 0.3	4.293 eV	4.294 eV

Table 4-1. Table of Theoretical and Experimental Bandgap Energies of $Al_xGa_{1-x}N$

	Theoretical Bandgap Energy	Experimental Bandgap Energy
GaN	3.476 eV	3.479 eV

Table 4-2. Table of Theoretical and Experimental Bandgap Energies of GaN

Cathodoluminescence and Photoluminescence spectra of Si implanted $Al_xGa_{1-x}N$

The CL and PL emission spectra were richly structured, resolved over a spectral range of 1.8 to 4.6 eV (7000 to 2500 Å) with identical excitation conditions. The resolution of the CL and PL measurements were 0.1 meV. Cathodoluminescence spectra taken at 5 K of Si implanted $Al_{0.1}Ga_{0.9}N$ annealed at 1350 °C for 120 seconds is shown in figure 4-1. The unimplanted/annealed sample has a donor bound exciton (D°,X) peak at 3.753 eV. The implanted samples have a (D°,X) peak that is red shifted from high to low dose. The peak values are 3.771, 3.755, and 3.699 eV for 1 x 10^{15}, 1 x 10^{14}, and 1 x 10^{13} ions per cm^2. The small differences between peak values of the (D°,X) peak is possibly due to slightly different amounts of aluminum incorporated into the $Al_xGa_{1-x}N$ crystal structure depending on the location of the test sample, in particular for a dose of 1 x 10^{13} ions per cm^2 . The two higher dose samples exhibit a green luminescence (GL) band at 2.473 eV. The GL band is most likely due to impurities or deep levels. No donor acceptor pair (DAP) peak is present. The highest dose sample has a GL peak that is more intense than the other doses. The peak at 1.85 eV for the lowest dose sample is due to secondary order effects and the abrupt change in intensity at 2.430 eV for the 1 x 10^{14} ions per cm^2 sample is due to the insertion of a low band pass filter. In all current spectra, small interference peaks appear due to a thin nitride epilayer on the sapphire substrate, in particular for broad bands. The annealing temperature seems sufficient to remove ion implantation damage, but a shorter anneal time might possibly produce better results.

$Al_{0.2}Ga_{0.8}N$ provided the most interesting results. Cathodoluminescence spectra

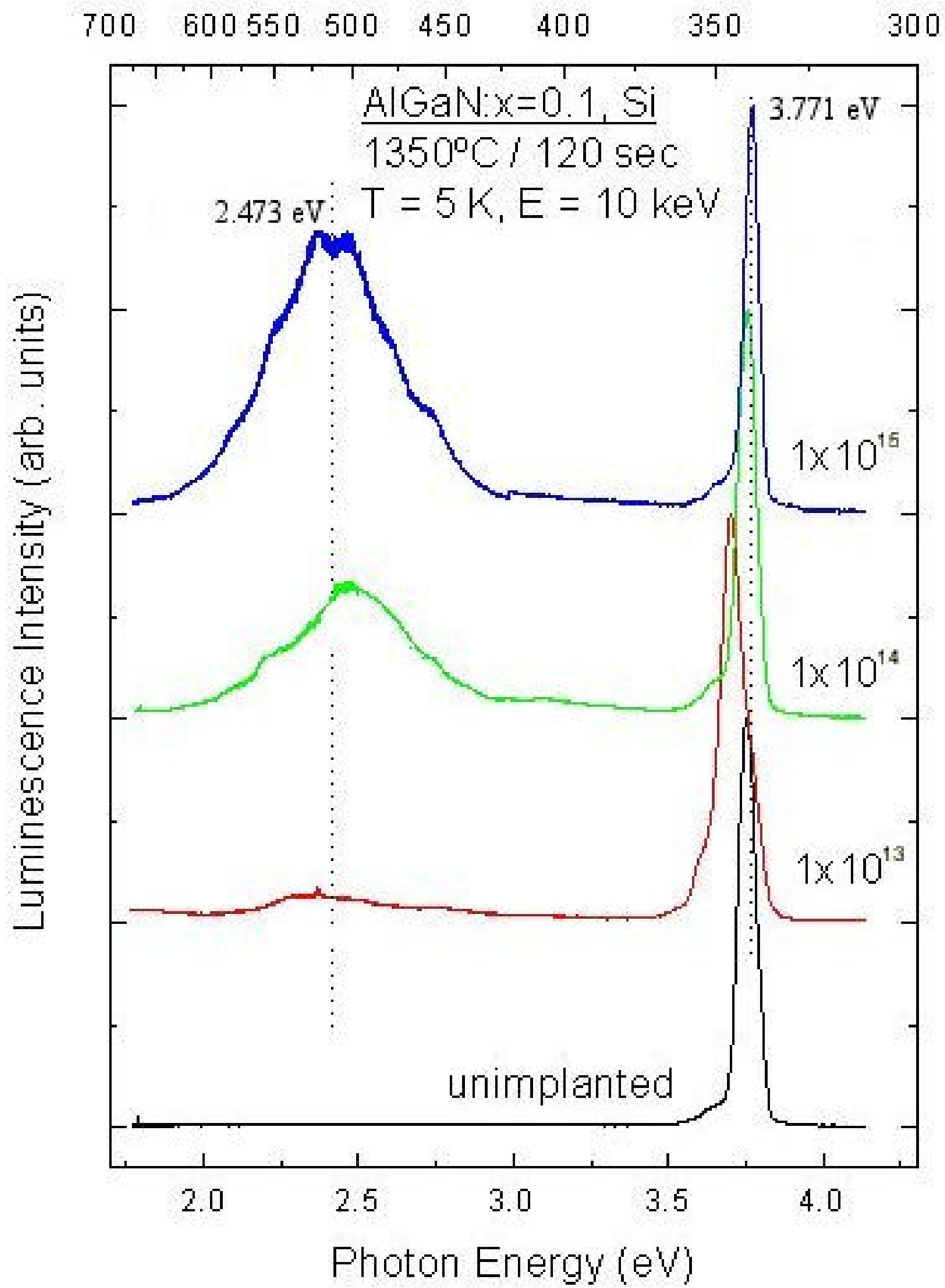

Figure 4-1. CL spectra taken at 5 K of $Al_{0.1}Ga_{0.9}N$ implanted with various doses of Si annealed at 1350 °C for 120 seconds

taken at 5 K of Si implanted $Al_{0.2}Ga_{0.8}N$ annealed at 1250 °C for 120 seconds is shown in figure 4-2. The (D°,X) peak is given at 4.008 eV for the unimplanted but annealed and 1 x 10^{14} sample. The peak to the right of the (D°,X) peak for these samples is a free exciton peak at 4.095 eV. The unimplanted sample also exhibits violet luminescence (VL) at 3.100 eV, blue luminescence (BL) at 2.703 eV, and peaks in both the yellow luminescence (YL) at 2.208 eV and the GL at 2.429 eV. This possibly indicates an abundance of impurities or deep levels. The sample with a dose of 1 x 10^{14} ions per cm^2 shows a broad band transition in the yellow at 2.298 eV and the highest dose sample shows a very broad band in the BL centered at 2.602 eV. The highest dose sample has a (D°,X) peak at 3.927 eV, which is at a much lower energy than the others. This particular spectrum is a PL measurement at 3 K. No shallow DAP peak is present in any sample spectrum. The annealing temperature seems insufficient to remove ion implantation damage because the (D°,X) peak is not sharp and there is an inconsistency of band transitions among the various doses of Si. Cathodoluminescence spectra taken at 5 K for various doses of Si implanted $Al_{0.2}Ga_{0.8}N$ annealed at 1300 °C for 120 seconds are shown in figure 4-3. The sample with a dose of 1 x 10^{14} ions per cm^2 does not have a (D°,X) peak, but the unimplanted/annealed and the 1 x 10^{13} ions per cm^2 samples have one at 4.018 eV with a free exciton peak at 4.098 eV. There appears to be second order effect peaks for the 1 x 10^{14} ions per cm^2 and the unimplanted sample. For the dose of 1 x 10^{14} ions per cm^2 a weak YL peak is present centered at 2.293 eV, and a very weak YL signal for the 1 x 10^{13} ions per cm^2 sample. The highest dose sample, which is a PL spectrum taken at 3 K, has a (D°,X) peak at 3.902 eV and a weak GL peak at 2.513 eV.

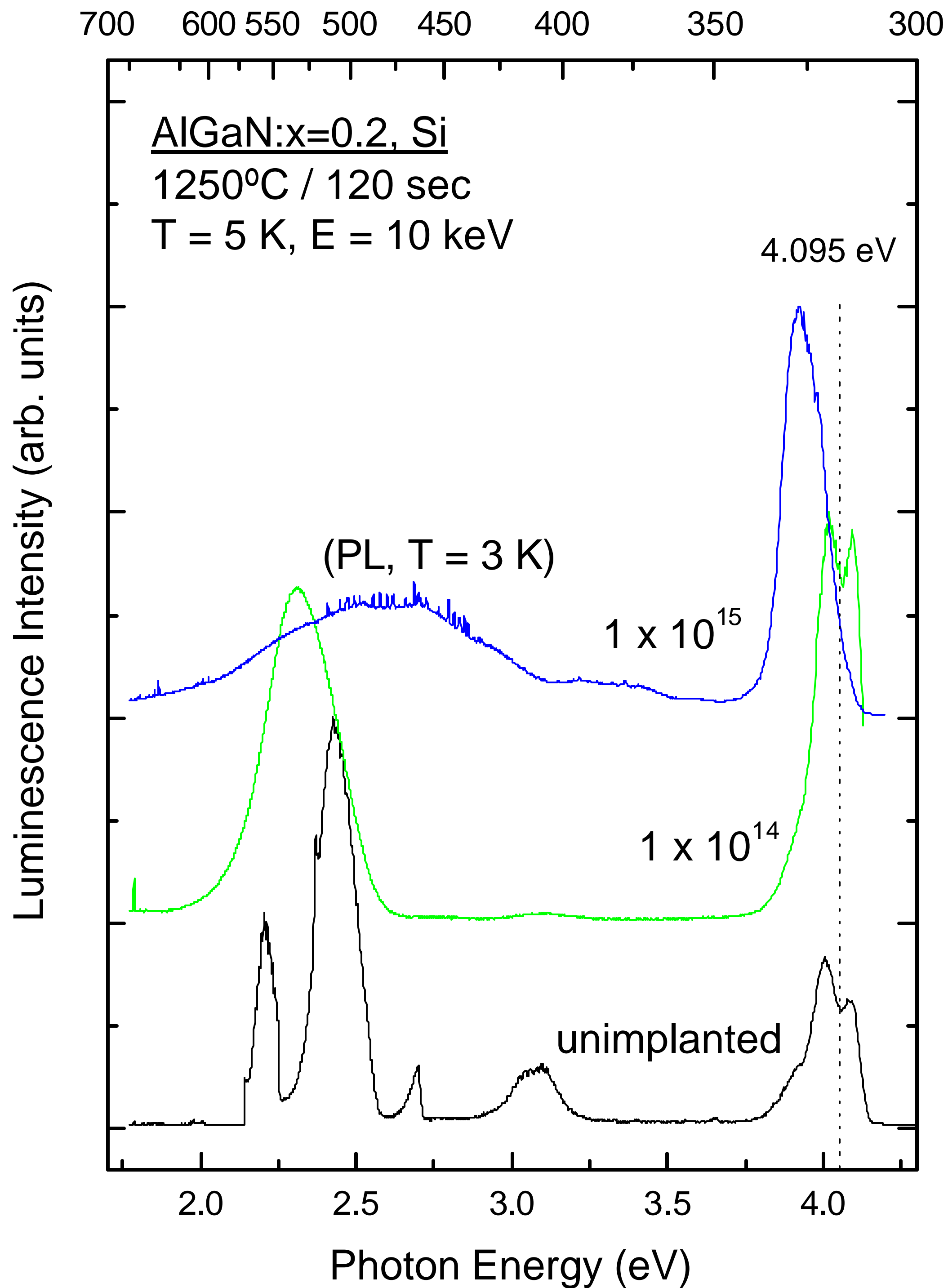

Figure 4-2. CL spectra taken at 5 K of $Al_{0.2}Ga_{0.8}N$ implanted with various doses of Si annealed at 1250 °C for 120 seconds (Note: PL for 1 x 10^{15} ions per cm^2 sample.)

No DAP peak is present for all samples. 1300 °C is an acceptable annealing temperature, but perhaps not the best choice as indicated by the results for 1350 °C anneal.

Cathodoluminescence spectra taken at 5 K for Si implanted $Al_{0.2}Ga_{0.8}N$ annealed at 1350 °C for 120 seconds is shown in figure 4-4. The unimplanted/annealed sample has a free exciton peak at 4.091 eV and a (D°,X) peak at 4.005 eV. It also has a very weak YL peak at 2.233 eV. The sample with a dose of 1 x 10^{13} ions per cm^2 has a free exciton and (D°,X) peak at 4.060 and 4.026 eV, respectively. A very weak VL peak is present centered at 3.125 eV, along with a very weak YL peak at 2.231 eV. The sample with a dose of 1 x 10^{14} ions per cm^2 has a free exciton peak at 4.082 eV and a (D°,X) peak at 4.042 eV. The sample with a dose of 1 x 10^{15} ions per cm^2 has a (D°,X) peak at 3.913 eV and a broad band BL centered at 2.628 eV. The weak free exciton peak and broad bands present in the samples annealed at 1350 °C along with the relatively sharp (D°,X) peak suggest this temperature is an appropriate one for annealing $Al_{0.2}Ga_{0.8}N$.

$Al_{0.2}Ga_{0.8}N$ implanted with 1 x 10^{15} ions per cm^2 produced excellent Hall effect measurements. The electrical activation efficiency was 8%, 56%, and 92% for annealing temperatures of 1250, 1300, and 1350 °C, respectively. Therefore, temperature dependent photoluminescence spectra were taken for annealing temperatures from 1200 to 1350 °C, as well as a comparison at 3 K for all annealing temperatures. Each sample was annealed for 120 seconds. Photoluminescence spectra taken at 3 K for 1 x 10^{15} Si ions per cm^2 implanted $Al_{0.2}Ga_{0.8}N$ annealed at various temperatures are shown in figure 4-5. The as-grown sample has a (D°,X) peak at 3.990 eV. For the implanted and annealed samples, the (D°,X) peak is at 3.912 eV, and the intensity increases with anneal

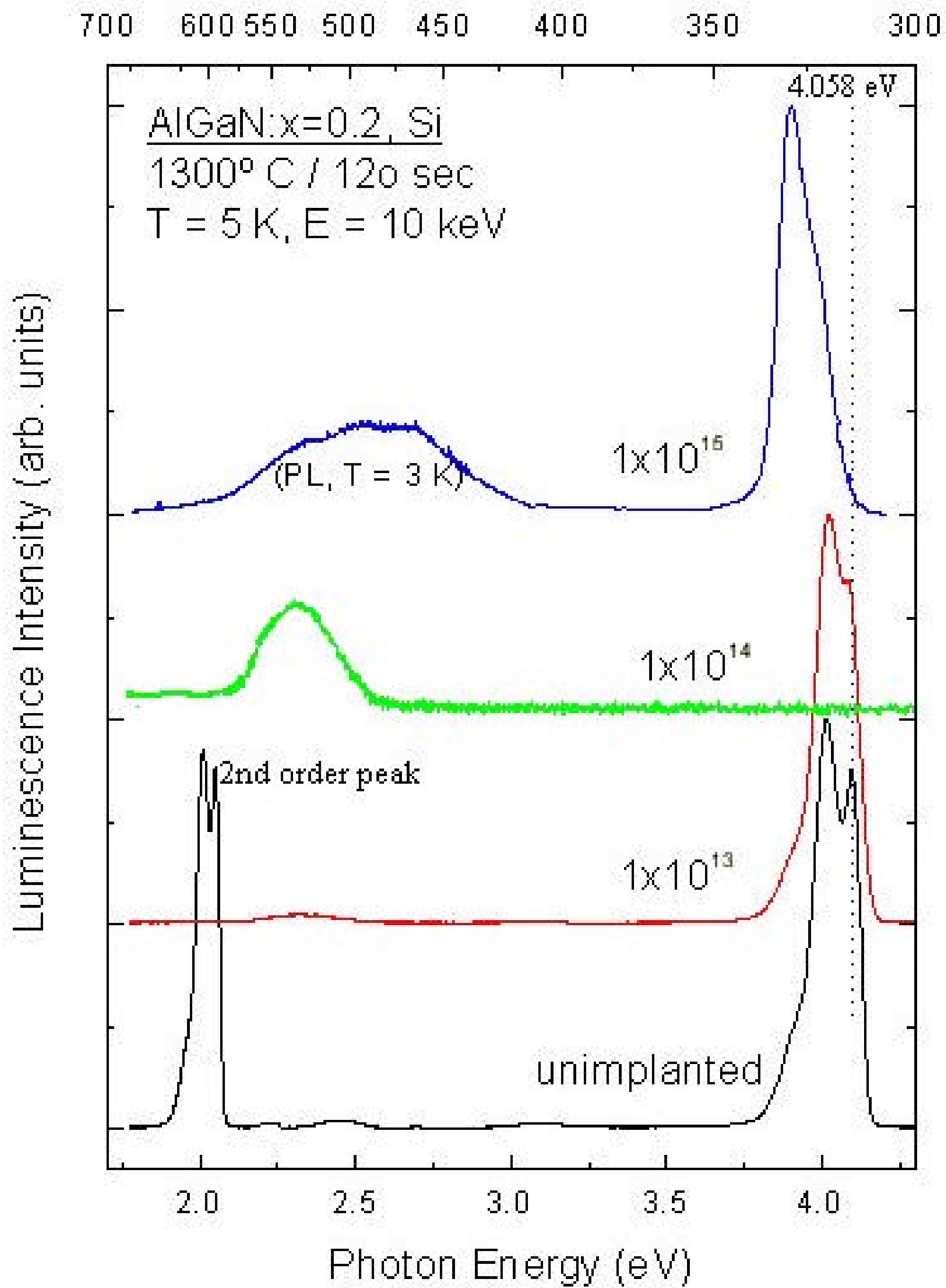

Figure 4-3. CL spectra taken at 5 K of $Al_{0.2}Ga_{0.8}N$ implanted with various doses of Si annealed at 1300 °C for 120 seconds (Note: PL for 1 x 10^{15} ions per cm^2 sample.)

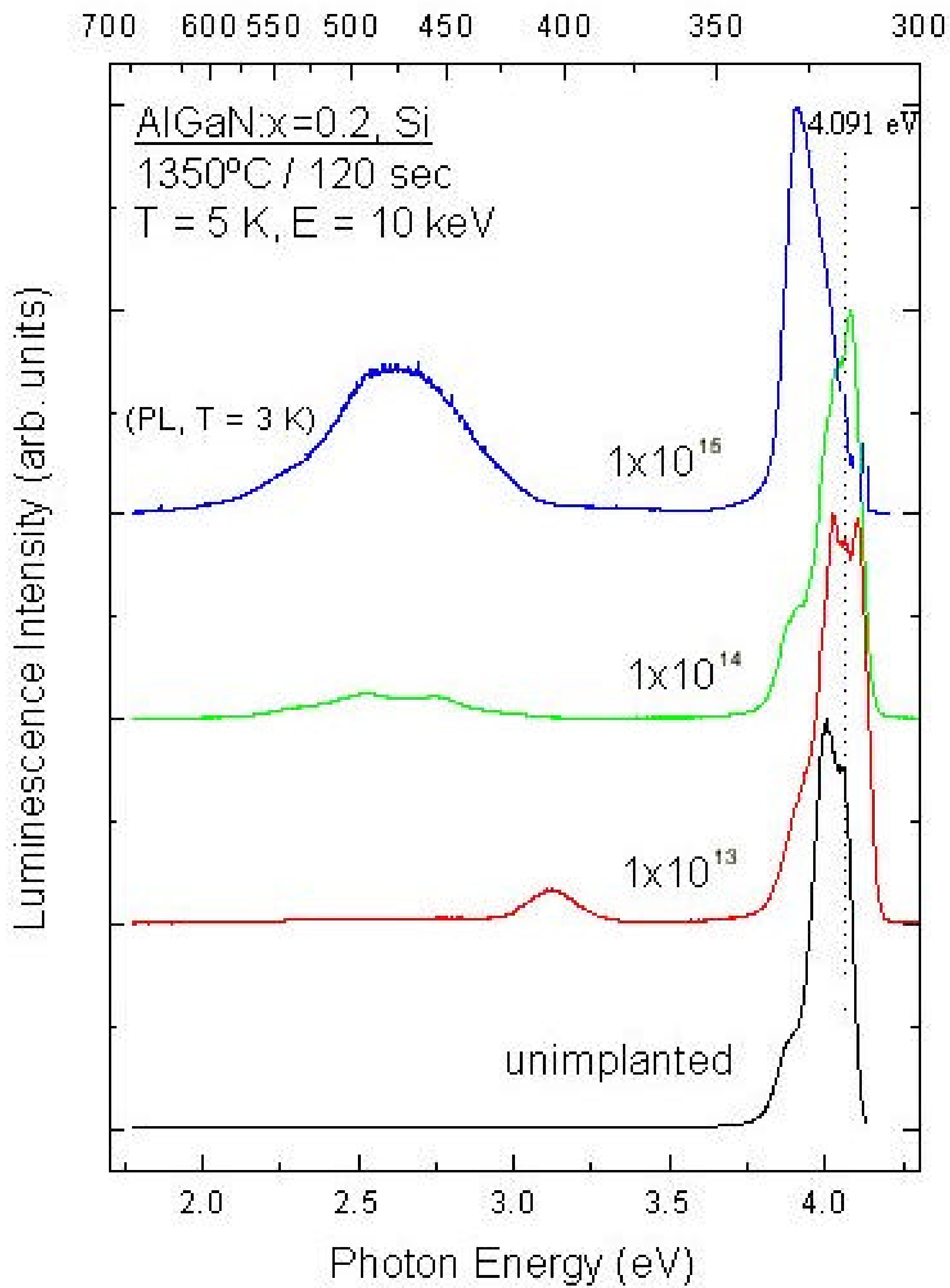

Figure 4-4. CL spectra taken at 5 K of $Al_{0.2}Ga_{0.8}N$ implanted with various doses of Si annealed at 1350 °C. (Note: PL of 1 x 10^{15} ions per cm^2 sample.)

temperature. For the samples annealed at 1200 °C, there is a GL peak at 2.363 eV. At all other anneal temperatures there is a broad BL band peak centered at 2.610 eV. No shallow DAP peak is present in any samples. The spectra for the unimplanted sample at 1350 °C aligns well with the as-grown sample, implying no significant damage due to the annealing process.

Temperature dependent photoluminescence spectra taken from 3 to 300 K for 1 x 10^{15} ions per cm^2 of Si implanted $Al_{0.2}Ga_{0.8}N$ annealed at 1300 °C is shown in figure 4-6. There are three features prevalent in the spectra, a (D°,X) peak, a free exciton shoulder peak, and a BL band. At 3 K, the (D°,X) peak is at 3.915 eV. The free exciton peak is located at 4.098 eV, and the broad band BL peak is centered at 2.595 eV. As the temperature increases to 300 K, there is a red shift of the peak by 30 meV, and this is due to the bandgap decreasing as the temperature increases. As the temperature increases, the (D°,X) peak intensity gradually decreases, and it almost disappears at 300 K. However, the intensity of the BL band remained about the same.

Temperature dependent photoluminescence spectra taken from 3 to 300 K for 1 x 10^{15} ions per cm^2 of Si implanted $Al_{0.2}Ga_{0.8}N$ annealed at 1350 °C is shown in figure 4-7. The three features present are a (D°,X) peak, a free exciton shoulder peak, and a BL peak. At 3 K the (D°,X) peak is located at 3.915 eV. . The free exciton peak is located at 4.058 eV. The broad BL spectra is centered at 2.595 eV. Again, red shifting occurs and at 300 K the (D°,X) peak has shifted 30 meV. Ion implantation damage removal seems successful for the $Al_{0.2}Ga_{0.8}N$ samples annealed at 1350 °C, as evidenced by the figure.

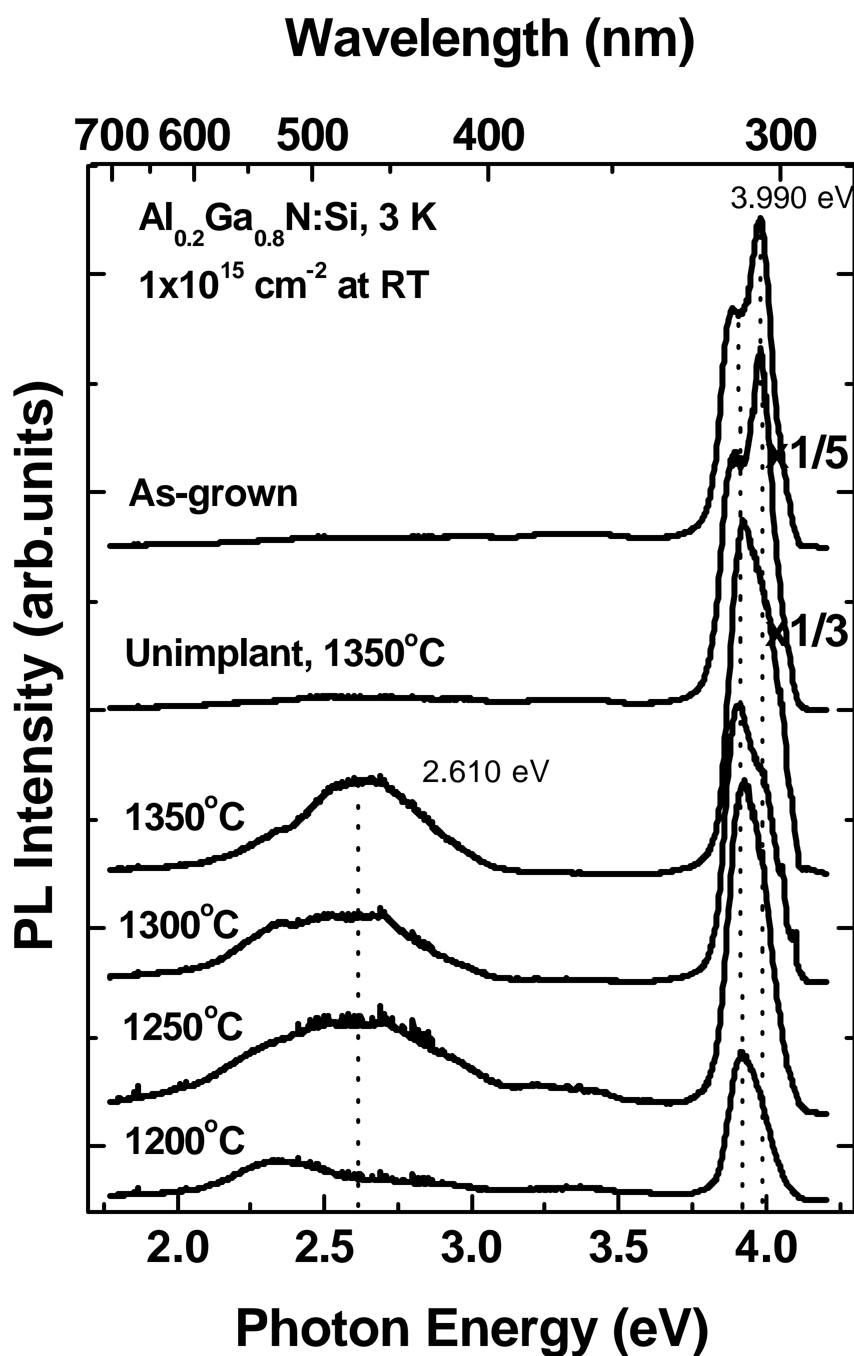

Figure 4-5. PL spectra taken at 3 K for $Al_{0.2}Ga_{0.8}N$ implanted with 1 x 10^{15} Si ions per cm^2 annealed at various temperatures for 120 seconds

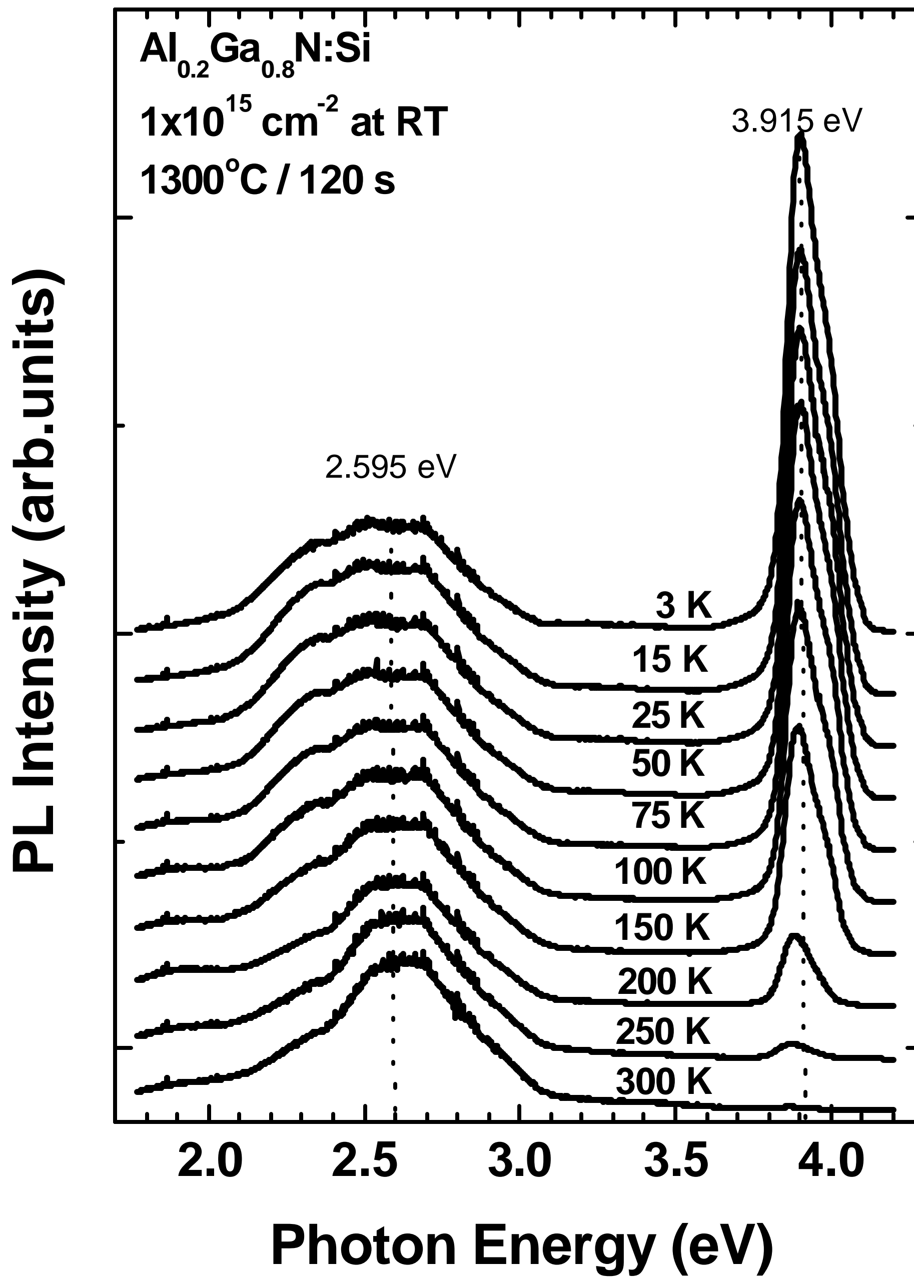

Figure 4-6. Temperature dependent PL spectra for $Al_{0.2}Ga_{0.8}N$ implanted with 1 x 10^{15} Si ions per cm^2 annealed at 1300 °C for 120 seconds

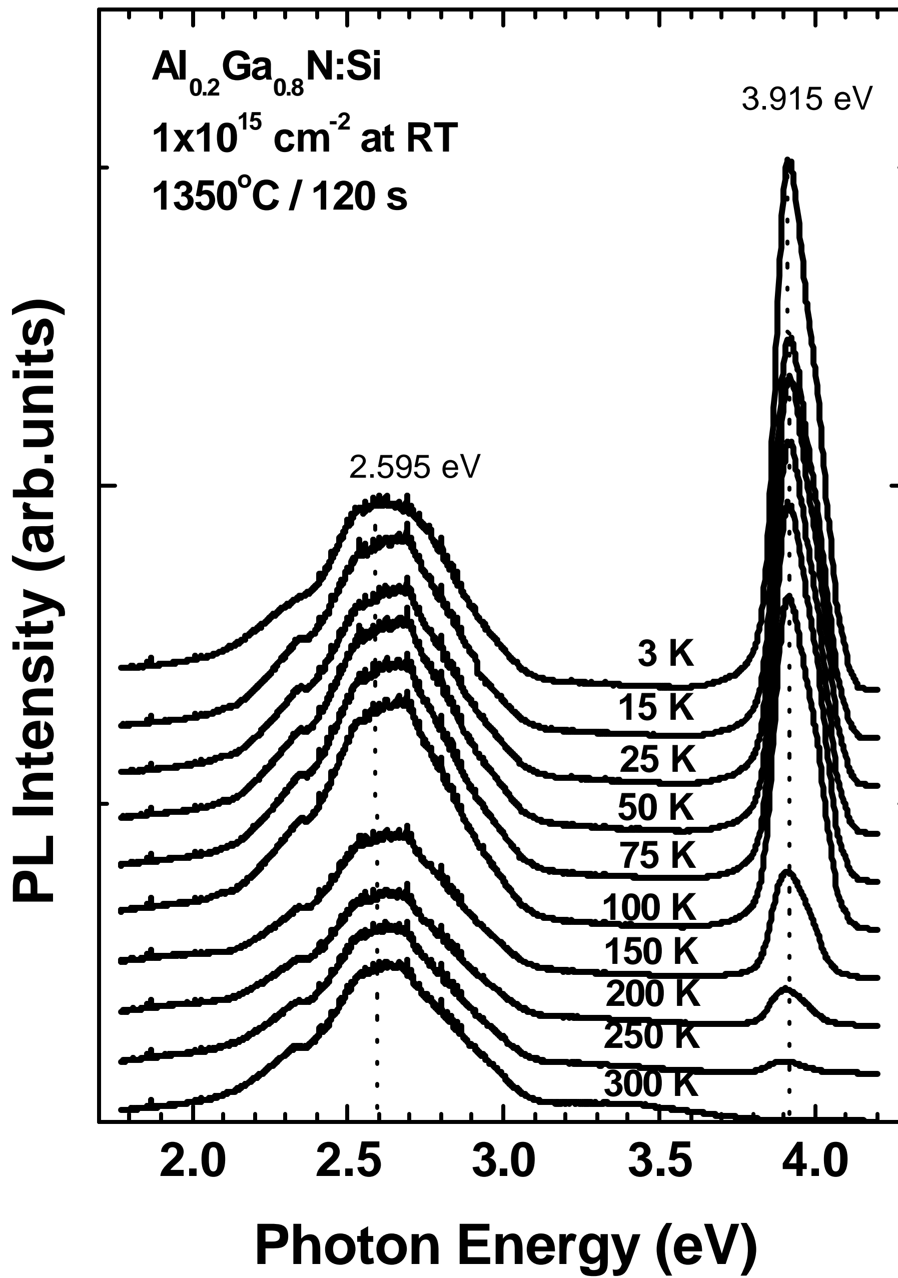

Figure 4-7. Temperature dependent PL spectra for $Al_{0.2}Ga_{0.8}N$ implanted with 1 x 10^{15} Si ions per cm^2 annealed at 1350 °C for 120 seconds

Cathodoluminescence spectra of Mg implanted GaN

Cathodoluminescence spectra obtained at 5 K from Mg doped GaN implanted with various doses of Mg at 25 ºC at 200 keV annealed at 1350 ºC for 30 seconds are shown in figure 4-8. No (Dº,X) peak is present in any of the implanted and/or annealed samples. The unimplanted Mg doped GaN shows a (D°,X) peak at 3.479 eV, and a DAP peak at 3.286 eV due to isolated acceptors and residual donors is observed for the as-grown and unimplanted/annealed samples along with its phonon replicas. The first LO phonon peak is at 3.180 eV and the second LO phonon peak is at 3.090 eV with a phonon energy of 95 meV. The broadening of the DAP peak on the lower energy end is possibly due to higher order phonon replicas. All Mg implanted samples show no DAP peak, suggesting that the samples may be fully compensated. Different annealing temperatures and times might be required. The dominant peak in the implanted samples is a green luminescence (GL) band at 2.370 eV, which was seen in all Mg-implanted GaN in previous research (Fellows, 2001). The GL peak is attributed to Mg induced defect clustering or residual implantation effects. The GL peak shows about the same intensity in all implanted samples. From the literature, Mg implanted GaN whose PL spectrum exhibited GL bands has shown no p-type conductivity.

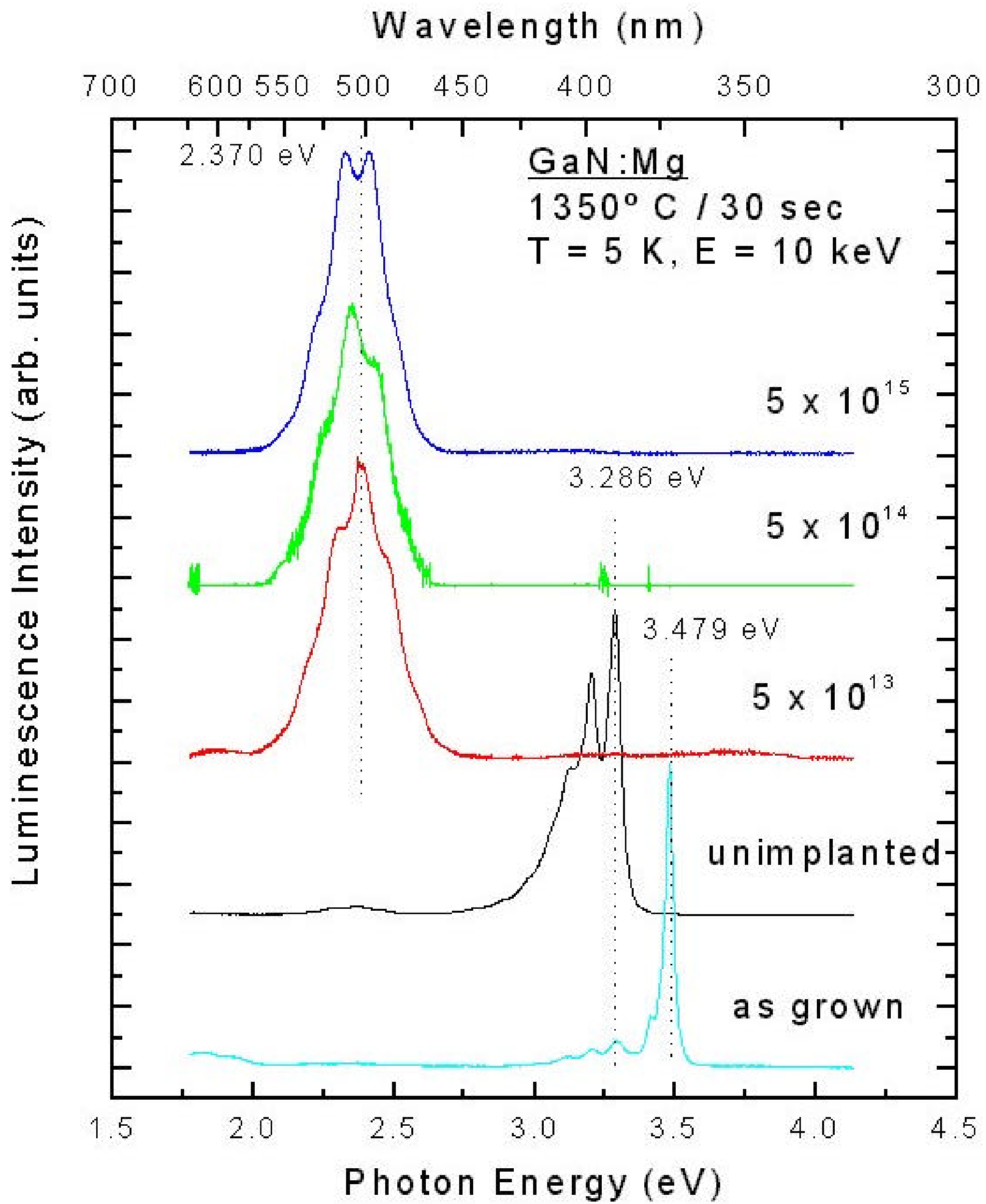

Figure 4-8. CL spectra taken at 5 K of p-type (Mg doped) GaN implanted with various doses of Mg annealed at 1350 °C for 30 seconds

Cathodoluminescence spectra of GaN co-implanted with Si and N

Cathodoluminescence spectra taken at 5 K from GaN co-implanted at 25 ºC with Si and N at 200 keV and annealed at 1250 ºC is shown in figure 4-9. For the as-grown and the lowest dose of 1 x 10^{13} Si / 9 x 10^{12} N ions per cm^2, there is a (Dº,X) peak at 3.479 eV. This peak is not present in the higher doses, but the origin of the peak is unknown at the present. Another peak is present at 3.420 eV in the 1 x 10^{14} Si/9 x 10^{13} N ions per cm^2 spectra. The DAP peak with phonon replicas is at 3.290 eV. This peak is strongest in the lowest dose. This may be due to the fact that less damage is caused by lower dose ion implantation, and therefore recovery is easier at lower anneal temperatures for lower doses. There is a broad band extending from 2.0 – 2.6 eV centered at 2.220 eV, and this is the yellow luminescence (YL) peak. This YL peak is due to native defects or impurity related complexes, and the intensity depends on the concentration of the impurities present. The as-grown (unannealed) sample has a very sharp (D°,X) peak. The high dose implanted samples do not all have a sharp peak that closely resembles the as-grown peak, so the annealing temperature may not be sufficient to remove ion implantation damage.

Cathodoluminescence spectra taken at 5 K from GaN co-implanted with Si and N annealed at 1300 ºC is shown in figure 4-10. The sharp peak at 2.38 eV is an effect from the electron gun and is not related to the luminescence data. A very weak (Dº,X) peak is ascertainable at 3.479 eV in the spectra for 1 x 10^{13} Si / 9 x 10^{12} N ions per cm^2 sample. The DAP peak is at 3.290 eV and its phonon replicas of the as grown sample which are shown in the figure. The DAP peak appears very weakly in the implanted samples,

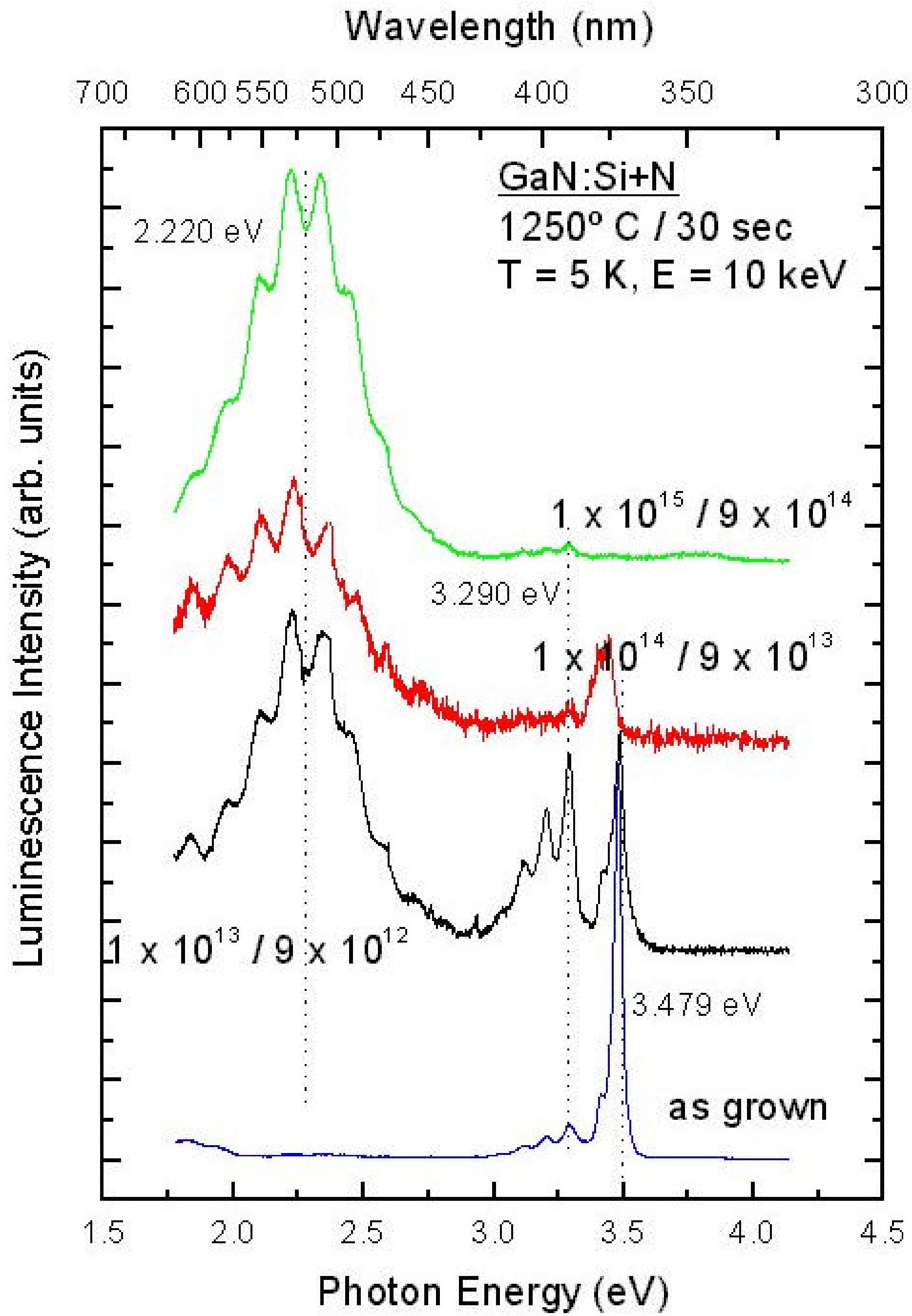

Figure 4-9. CL spectra taken at 5 K of GaN implanted with various doses of Si and N annealed at 1250 °C for 30 seconds

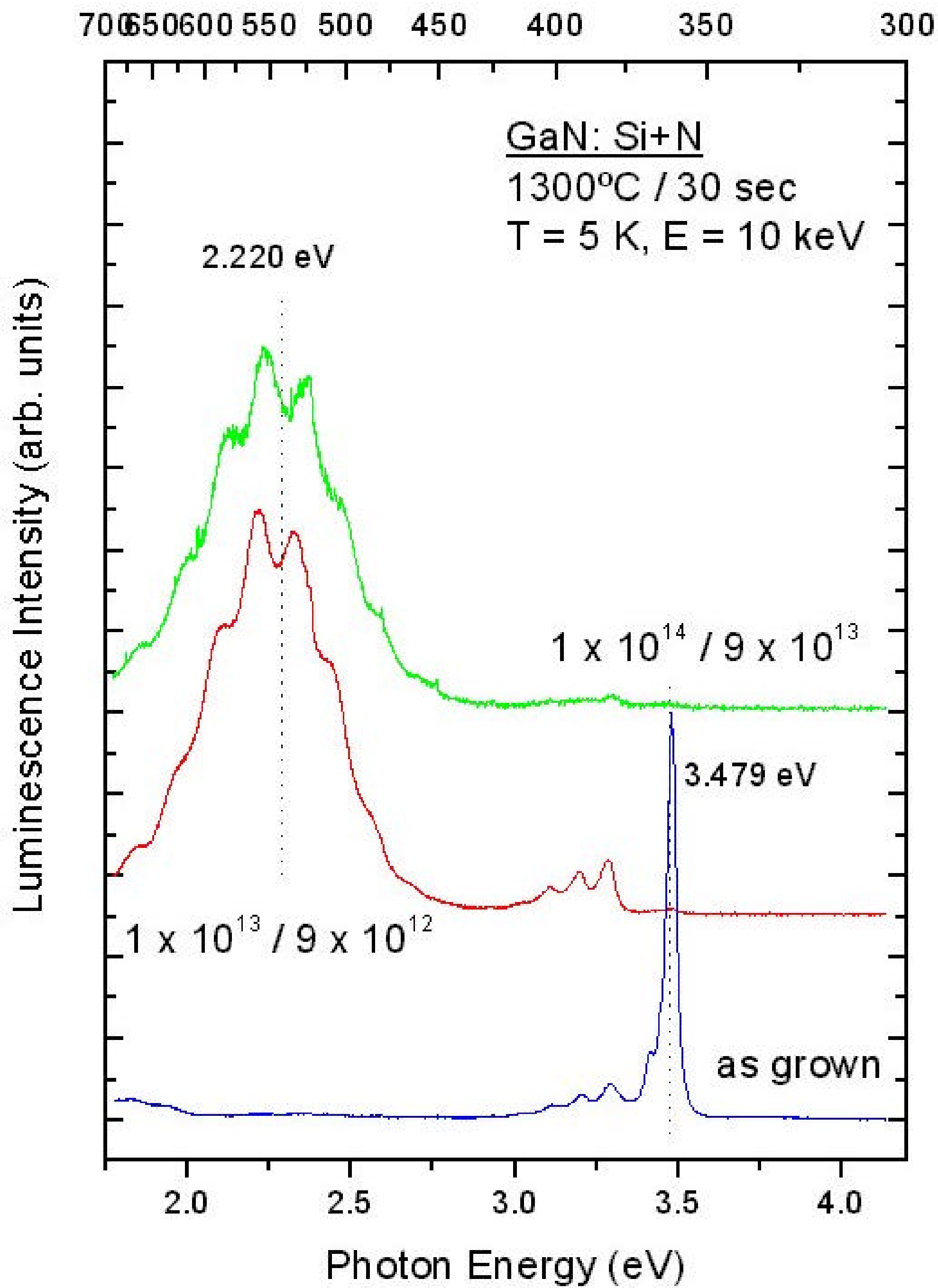

Figure 4-10. CL spectra taken at 5 K of GaN implanted with various doses of Si and N annealed at 1300 °C for 30 seconds

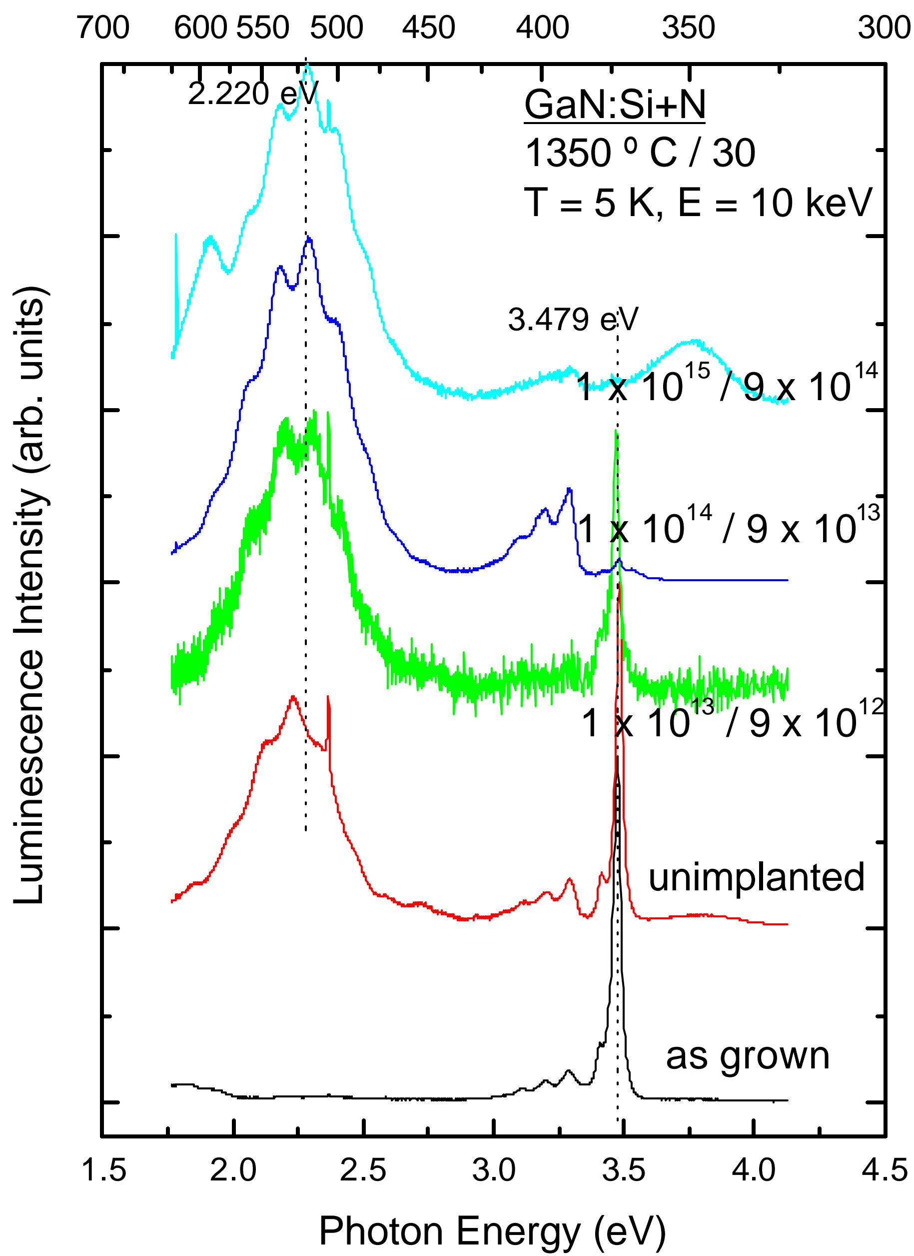

Figure 4-11. CL spectra taken at 5 K of GaN implanted with various doses of Si and N annealed at 1350 °C for 30 seconds

indicating that full recovery from ion implantation damage may not be achieved at an anneal temperature of 1300 ºC. A broad YL peak centered at 2.220 eV is present for all implanted samples

Cathodoluminescence spectra taken at 5 K from GaN co-implanted with Si and N and annealed at 1350 ºC is shown in figure 4-11. The (Dº,X) peak obtained from unimplanted and annealed sample of GaN is very sharp at 3.479 eV, indicating no significant damage created during the anneal. The (Dº,X) peak reduces in intensity as the ion dose increases, almost disappearing at the highest dose of 1 x 10^{15} Si / 9 x 10^{14} N ions per cm^2. The DAP is present in all doses and the unimplanted sample at 3.290 eV with its phonon replicas. The sample with the ion dose of 1 x 10^{13} Si / 9 x 10^{12} N had a poor surface quality and showed a very weak DAP luminescence. The DAP peak intensity is strongest for a dose of 1 x 10^{14} Si / 9 x 10^{13} N ions per cm^2 and becomes very weak for a dose of 1 x 10^{15} Si / 9 x 10^{14} N sample. The ion implantation damage appears considerably removed at 1350 ºC for 1 x 10^{14} Si / 9 x 10^{13} N but not for the highest dose. A strong YL peak was present in all samples centered around 2.220 eV.

V. Conclusions and Recommendations

The primary objective of this research was to perform a comprehensive and systematic optical activation study of GaN and $Al_xGa_{1-x}N$ implanted with various doses of Si ions as a function of implantation dose and annealing temperature. The goal of this research was to increase the understanding and ability to use GaN and $Al_xGa_{1-x}N$ in enhanced optical device applications.

$Al_xGa_{1-x}N$ (x = 0.1, 0.2, 0.3) wafers capped with 500 Å AlN were implanted at room temperature with 200 keV Si ions at doses ranging from 1 x 10^{13} to 1 x 10^{15} ions per cm^2 and annealed from 1200 to 1350 °C for 120 seconds. PL and CL spectra were collected at low temperature, along with temperature dependent PL measurements for $Al_xGa_{1-x}N$ (x = 0.2). Wafers of both undoped and p-type GaN capped with 500 Å AlN were implanted at room temperature and annealed at temperatures ranging from 1250 to 1350 °C. Undoped GaN was implanted with 200 keV of Si and N ions at doses ranging from 1 x 10^{13} Si / 9 x 10^{12} N to 1 x 10^{15} Si / 9 x 10^{14} N ions per cm^2. P-type GaN wafers doped with Mg were implanted with various doses of Mg ranging from 5 x 10^{13} to 5 x 10^{15} cm^{-2}.

The results of CL and PL show that the most appropriate anneal temperature to remove ion implantation damage was 1350 °C for $Al_xGa_{1-x}N$ (x = 0.1, 0.2, 0.3). $Al_{0.2}Ga_{0.8}N$ samples gave excellent Hall effect measurements, and therefore a more extensive study was conducted using temperature dependent PL measurements for annealing temperatures of 1200 to 1350 °C. In general, $Al_xGa_{1-x}N$ exhibited a GL band.

A higher annealing temperature might produce better results for higher mole fractions of $Al_xGa_{1-x}N$.

Mg implanted p-type GaN showed no DAP peak, suggesting that the are most likely fully compensated or electrically inactive. However, a GL peak was present, most likely due to a Mg induced defect clustering.

GaN co-implanted with Si and N recovered well from ion implantation damage after a 1350 °C anneal. Most implanted samples exhibited a YL band centered around 2.2 eV.

Results from this research may help in understanding ion implantation damage recovery and therefore help in furthering the research to use GaN and $Al_xGa_{1-x}N$ in optoelectronic devices.

Appendix A

Publication Summary

The following is a complete list of conference proceedings and publications to be submitted resulting from this research.

Conference Proceedings

Luminescence studies of $Al_xGa_{1-x}N$ *implanted with Si*. E. Claunch, Mee-Yi Ryu, E. Chitwood, Y. K. Yeo, R. L. Hengehold, and T. Steiner. Submitted to the American Physical Society Meeting, March 3-7, 2003 held in Austin, Texas.

Luminescence studies of $Al_xGa_{1-x}N$ *implanted with Si*. E. Claunch, Mee-Yi Ryu, E. Chitwood, Y. K. Yeo, R. L. Hengehold, and T. Steiner. To be submitted to the 5th International Conference on Nitride Semiconductors (ICNF-5), May 25-30, 2003 held in Nara, Japan.

Publication to be Submitted

Luminescence studies of $Al_xGa_{1-x}N$ *implanted with Si*. E. Claunch, Mee-Yi Ryu, E. Chitwood, Y. K. Yeo, R. L. Hengehold, and T. Steiner. To be submitted to the Applied Physics Letters.

Appendix B

Sample Cutting and Cleaning Procedures

Cleanroom:

1. Turn solvent hood hotplate ON and set to 100 ^{o}C.
2. Place clean 3" Si wafer face-up on hotplate.
3. Place as many 1¼" ceramic cutting disks face-down on hotplate as you have source wafers to cut.
4. Label a glassine envelope for each 5 mm x 5mm sample you expect to cut.
5. Place source wafer on appropriate-sized spinner vacuum chuck, spin wafer and clean with acetone, methanol, and blow dry w/N_2.
6. Cover entire surface of source wafer with 1813 photoresist using dropper and spin on at 4000 rpm for 30 sec.
7. Place PR covered wafer face up on Si wafer for 4 min.
8. At 4 min mark, flip source wafer over and melt crystal bond onto backside ensuring edge from which samples will be cut has full coverage. Note: avoid getting crystal bond on Si wafer—this will make the mounting process go much smoother.
9. Immediately touch the face of a 1¼" ceramic cutting disk to the crystal bond coated wafer surface and lift. If wafer is stuck to Si wafer, carefully loosen with tweezers and reattempt bonding. If wafer bonds to disk, set aside to cool. Uniquely identify each ceramic disk. *** **Write down which disk has which source wafer!!!** ***
10. Repeat steps 5-9 for each source wafer to be cut.
11. Place all mounted wafers in transport box along with tweezers, crystal bond, and the brass mounting ring.
12. Turn OFF hotplate and remove Si wafer.
13. Place Si wafer on appropriate-sized spinner vacuum chuck, spin wafer and clean with acetone, methanol, blow dry w/N_2.

Wire saw:

14. Carry box to wire saw room and turn on hotplate in fume hood on very lowest temperature.
15. Ensure there is adequate distilled drip water for wire saw.
16. Securely mount a disk on the wire saw pedestal.
17. Align wire parallel to sample edge to be cut so that wire is just barely not touching sample.
18. Once wire and sample are properly aligned, activate and zero the sliding micrometer.
19. Lower saw swing arm and slide sample tray until micrometer reads ± 5.18 mm.
20. Set appropriate drip rate; position drip spot over sample and wire.
21. Turn ON saw motor, and set to 50%.
22. Cut sample slice. When wire is almost through sample minimize pressure between sample and wire to prevent uneven cutting or chipping of sapphire substrate.
23. Lower swing arm, turn saw to 0%.
24. Secure swing arm, remove ceramic disk and place face-up on hotplate until the next wafer has been cut.
25. Repeat steps 16-24 for next source wafer to be cut.
26. Once crystal bond has been sufficiently softened on previously cut wafer, carefully remove the portion of source wafer that will not be cut into samples, remembering its ID.

27. Repeat steps 16-23 for each strip that will be cut into square samples; make initial wire alignment with the end of the strip that is closest to the center of the grown wafer—in some cases this requires remembering the location of the wafer flat.
28. After each square sample is cut, zero the micrometer and slide the sample tray ± 5.18 mm until end of strip is reached; continue until all samples are cut, keeping careful track of which pieces belong to which wafer.
29. Lower and secure saw swing arm, turn OFF saw; turn OFF hotplate.

Cleanroom:

30. Return to cleanroom and turn solvent hood hotplate ON and set to 100 ^{o}C; Place all cutting disks face-up on hotplate
31. Set out as many 2" diameter petri dishes with covers as you have source wafers and fill each ~? full w/ acetone.
32. Once crystal bond has been sufficiently softened, carefully remove each sample and place all pieces from each source wafer in their own petri dish ***** Organization is crucial to keeping track of which samples are which! *****
33. Once all the pieces are soaking in covered dishes of acetone, turn OFF the hotplate.
34. Add DI water to each dish with a ratio of acetone:DI of about 3:1 to aid in removing the ceramic disk residue.
35. Fill the ultrasonic cleaner with DI water to the level of fluid in each petri dish.
36. Place each dish in the ultrasonic cleaner simultaneously <u>ONLY</u> if you can tell them apart and ultra for 20 seconds.
37. Remove dishes from the cleaner; carefully flush each dish with clean acetone and cover.
38. Clean each piece one at a time—holding with tweezers, rinse with acetone, methanol, and DI water, blowing dry w/N2 and immediately placing in the appropriate glassine envelop prepared beforehand.
39. Once all the samples and remaining source wafers (and remnants) have been cleaned and packaged, clean all quartzware with acetone and methanol--wiping will likely be necessary due to the PR, crystal bond and ceramic residue.

Appendix C

Oxy-Gon Sample Preparation Procedures

1. Select all the 5 mm x 5mm samples (which were previously cut & cleaned) you'll anneal.
2. On the cleanroom table, place a clean 3" Si wafer on a cloth wipe.
3. While holding them face down on the Si wafer, uniquely scribe the Al_2O_3 backside of each sample type (e.g., " / ", " < ", " | ", "O", "L", etc.)
4. Logically (e.g. hot and cold of same dose) and physically (e.g., best size match) pair up the samples.
5. After scribing, place all samples to be annealed in a 2" petri dish for temporary storage and transport.
6. Measure out ~ 1¾" of Ta-wire for each sample pair to be annealed, cutting one long piece.
7. Place this single piece of Ta-wire in a 2" petri dish, submerge the wire in TCE, and cover the dish.
8. Clean all samples again (front and back) with acetone and methanol rinses (and, as necessary to fully remove ceramic disk residue and make each surface mirror-like, DI H_2O), blow dry with N_2.
9. Rinse and flush the Ta-wire in the dish with acetone, then methanol. While still wet, pull the wire between a clean cloth wipe to dry.
10. Cut the wire into 1¾" sections.
11. Identify which samples will be wrapped face-to-face noting which sample will be on top.
12. Note: Practicing this process several times on a pair of junk samples is recommended.
13. Place a piece of Ta-wire on a cloth wipe and center the face-to-face samples in the middle of the wire.
14. Using two sets of tweezers, press the center of the samples together while wrapping the ends of the wire up and back across the top of the samples.
15. Bend first one end of the wire 90° at the center of the samples across the other wire, then bend the other end 90° so they interlock (like string on a Christmas package, or twine on a bail of hay).
16. Flip the sample pair over and repeat steps 14-15.
17. Flip the sample pair over and repeat steps 14-15 again, at which point you should have just enough wire to complete the final bends (step 15), only here interlock the ends and bend 180° vice 90°.
18. Keep the samples fully face-to-face throughout the process and wrap them securely. Any uncovered regions along the edge will be destroyed by the anneal—the samples must overlap perfectly!
19. Place the sample pair in the glassine envelope of the sample that started (and finished) on top.
20. Repeat steps 13-19 for each sample pair.
21. Place all the glassine envelopes in a plastic box and double bag for transport to anneal furnace.
22. Be sure to bring tweezers, gloves, wipes, and a metal dish for transport to/from the furnace.

Oxy-Gon AlN/GaN Anneal Procedures

System Start-Up (process selection switch should be in **STANDBY**):

1. Turn **ON** 80 psig house air (vent and vacuum valves are air pressure activated)
2. Turn **ON** Main Power switch (handle on lower front panel)
3. Turn Roughing pump **ON** (green button); Roughing pulls on Turbo to 10^{-3} Torr
4. Turn Turbomolecular pump **ON** (green button); Turbo pulls on itself
5. If chamber is still under a vacuum, turn process selection switch to **VENT GAS**, else GOTO 8
6. Turn **ON** low-O_2 N_2 at tank and regulator; ensure ball valve on furnace works
7. When chamber reaches atmospheric pressure, Turn **OFF** low-O_2 N_2 or Ar at tank and regulator
8. **Open** chamber, propping door open with ruler as necessary
9. **Inspect** graphite elements and electrodes for discoloration/wear; carefully **wipe** off any condensation or residue from walls
10. **Remove** previous samples as necessary and **insert** new samples evenly spaced in center of puck, then **secure** chamber door

Soft Bake (to remove impurities from hot zone):

11. Turn process selection switch to **STANDBY**, then **ROUGH**
12. Turn **ON** Ion Gauge Controller to read TCs
13. Rough pump until chamber is mid 10^{-2} Torr (read TC2); this will take a few minutes; Turbo still pulls on itself, and pressure increases slightly at TC1
14. Turn process selection switch to **HI VACUUM** (Turbo pulls on chamber, Rough pulls on Turbo—TC2 drops quickly; TC1 increases then drops more slowly)
15. To remove trapped O_2 between GaN samples, **Repeat** steps 5 through 7 only, and begin again at step 11
16. Turn **ON** Ion Gauge Filament when TC2 is in the 10^{-3} Torr range, & pump until mid-high 10^{-5} Torr
17. After ~10 min into 2nd Hi-vac pull, **Open** H_2O inlet and outlet hand valves (only when chamber under vac. or filled with N_2)
18. Ensure yellow H_2O handles are open: 2, 4 & Chamber Main at 45°, 1 & 3 at full open, and H_2O safety light is ON.
19. **RESET** Over Temperature Controller to start pre-anneal soft bake of elements and chamber
20. Ensure Vacuum Interlock Bypass is OFF, Turn Heat Zone **ON**
21. Approx. 5 min after H_2O on, ion gauge should be mid-hi 10^{-5} Torr; **Ramp Up** AUTO/MAN power controls to 20%
22. Soft Bake chamber at 20% for 12 min (expect T to be approx. 230 ^{o}C; if significantly less, TC may be bad—abort run ***)
23. **Ramp Down** AUTO/MAN power controls to 8% and continue soft bake for another 5 min
24. **Ramp Down** AUTO/MAN power controls to 0%
25. Turn **OFF** Ion Gauge Filament (Heat Zone may go off simultaneously) ensuring P ~mid-hi 10^{-5} Torr

Set-Up Anneal Environment:

26. Turn **ON** low-O_2 N_2 or Ar at tank and regulator
27. Turn process selection switch to **VENT GAS** to backfill chamber to ~1 PSIG
28. Adjust vent floating ball valve so that chamber gas is just barely flowing

Anneal Process:

29. Turn **ON** Vacuum Interlock Bypass (and turn **ON** Heat Zone as necessary)
30. **Ramp Up** AUTO/MAN power controls for desired temperature profile (55% initially for graphite to minimize excessive current, then gradually up to 69% max at $T \geq 1150\ ^oC$ to maximize ramp rate). *Carefully* control power to not overshoot.
31. ANNEAL GaN samples
32. At appropriate time, **Ramp Down** AUTO/MAN power controls to 0%
33. Using floating ball valve, gently increase vent gas flow rate to the 10-15 lpm range
34. Turn **OFF** Heat Zone 10 min into cool-down (after power supply has cooled) on last run
35. Turn **OFF** Turbo during last run of the day
36. **Close** H_2O inlet and outlet hand valves, respectively, when $T<100\ ^oC$
37. When chamber reads $T<30\ ^oC$ (for AlN/GaN), **Close** floating ball valve, Turn **OFF** low-O_2 N_2 at tank and regulator
38. If annealing more samples, GOTO 8
39. **Open** chamber, propping door open with ruler as necessary, carefully **remove** samples, then **secure** chamber door

System Shut Down:

40. Turn process selection switch to **STANDBY**, then **ROUGH**
41. Rough pump until TC2 ~ mid 10^{-2} Torr, if Turbo is still on, Turn process selection switch to **HI VACUUM** until TC2 ~10^{-3}
42. If short term shut down (< 1 day), Turn process selection switch to **STANDBY**, Turn **OFF** Turbo, GOTO 45
43. Turn process selection switch to **VENT GAS**, and **Open** floating ball valve to backfill chamber to ~1 PSIG
44. **Close** floating ball valve; Turn process selection switch to **STANDBY**
45. Turn **OFF** vent gas at tank and regulator
46. Turn **OFF** Vacuum Interlock Bypass
47. Turn **OFF** Roughing pump
48. Turn **OFF** Ion Gauge Controller
49. Turn **OFF** Main Power switch
50. Turn **OFF** 80 psi house air

Appendix D

Post-Anneal Contact Preparation Procedures

1. Obtain HCl and HNO_3 acids and place within the acid fume hood.
2. Turn ON one solvent fume hood hotplate and set to 140 ^{o}C.
3. Turn ON the second solvent fume hood hotplate and set to 90 ^{o}C.
4. On the cleanroom table, place an annealed Ta-wire-wrapped sample pair on a clean cloth wipe.
5. Using two sets of tweezers, carefully break off the brittle Ta wire-wrap, keeping track of which sample is which throughout the process.
6. Visually examine the AlN surface for signs of Ga droplets, cracking/peeling etc. (A good AlN surface post anneal will be as mirror-like as when it was wrapped.)
7. If the identifying scribe markings on the backside are no longer clearly distinguishable (at any angle or over a reflective Si wafer) place the sample on a clean 3" Si wafer and re-scribe.
8. Place the samples in a 2" petri dish for temporary storage and transport.
9. Fill a clean 250 ml quartz beaker with 50 ml of DI H_2O; cover and place on the 140 ^{o}C hotplate.
10. Repeat steps 4-8 for each sample pair you have annealed.
11. Weigh out 1.63 g of KOH pellets (86% KOH) and tightly close the double bag.
12. Quickly place all pellets into the beaker of hot DI H_2O as the pellets will begin to melt in air.
13. Stir with tweezers until all pellets are fully dissolved and cover the beaker. (Although the hotplate is set at 140 ^{o}C, the DI H_2O will not boil, typically reaching at most 95 ^{o}C.)
14. *** Note: ensure the evaporator is not in use before proceeding with any acid processing.
15. Measure out 30 ml of HCl and place in a clean 250 ml quartz beaker.
16. Measure out 10 ml of HNO_3 and add to the HCl; gently circulate and cover the aqua regia.
17. Process ONLY the good morphology samples as the first batch (< 5-10% total metallic Ga surface area is good). Process all other samples in the second batch.
18. Place each sample in the 0.5M hot KOH solution; starting a 5 min timer on the first sample.
19. Continue placing samples one at a time at the same rate in the KOH sequentially along the circumference of the beaker and cover when finished.
20. When the samples have only 1 min left in the KOH, bring the covered beaker of aqua regia to the solvent fume hood and place on the 90 ^{o}C hotplate.
21. At 5 min, remove the samples at the same rate and in the same order in which you inserted them.
22. As each sample is removed, place it into a large (600 ml) beaker of clean DI H_2O.
23. Carefully rinse the samples in the beaker by dumping most of the DI/adding clean DI, dumping/adding—taking care to not even come close to loosing any samples. Leave at most 1" of DI in beaker.
24. When the aqua regia just begins to boil, place the samples into the acid solution directly from the DI beaker; starting a 2 min timer on the first sample.
25. Continue placing samples one at a time at the same rate in the aqua regia sequentially along the circumference of the beaker and cover when finished.
26. At 2 min, remove the samples at the same rate and in the same order in which you inserted them.

27. As each sample is removed, place it into a large beaker of clean DI H_2O.
28. When all the samples are in the DI, cover the aqua regia and turn off the 90 ^oC hotplate.
29. At this point, you may need to rinse a green residue off the metal tweezers, wipe, rinse and blow dry with N_2
30. Carefully rinse the samples in the beaker by dumping most of the DI/adding clean DI, dumping/adding—taking care to not even come close to loosing any samples. Leave at most 1" of DI in beaker.
31. Holding with tweezers, agitate each sample in the DI, remove, blow dry with N_2 and place in a clean 2" petri dish.
32. Repeat steps 18-31 for the second batch as necessary using the same acid and base solutions.
33. On the cleanroom table, carefully mount all samples face down on the van der Pauw shadow mask on a clean cloth wipe.
34. Adjust and secure each sample by gently tightening mounting screws until all samples are positioned for contacts as much in the corners as possible.
35. Note: Each row on the mask is a different sized square; generally the largest two square rows are best.
36. *** Note: This is an iterative and tedious process as tightening one sample may loosen another. ***
37. When all samples are securely squared, vent the evaporator and carefully insert the mask.
38. Remember to change the microscope window slides, check metal levels, secure door and "process".
39. Turn off both hotplates; clean up all acids, bases, DI, Ta-wire-pieces, etc.

Edwards Auto 306 Evaporator Procedures

Sample preparation:
1. Degrease sample with solvents (acetone, methanol) DI rinse and N_2 blow dry.
2. Remove any oxides with 2 min of boiling aqua regia (HNO_3:HCl, 1:3), DI rinse and N_2 blow dry.

Vent chamber, Mount/Remove sample & Create vacuum:
1. Ensure chamber is not in use and has been cooled for at least ½ an hour after the last evaporation.
2. Press "**Seal/Vent**" and lift chamber clip—door will open *easily* at 7.6E2 Torr—not until!
3. When vented, **open** chamber door and remove sample jig—if removing, do so & go to Step 7.
4. **Mount** cleaned sample(s) properly on jig
5. Physically **verify** the metals in each carousel positions and note for assigning layer parameters.
6. Check amount of metal in hearths to be used and **fill only as necessary**—half full is OK.
7. **Insert jig** into chamber, and secure door.
8. Press "**Process**" to start vacuum.
9. **Fill** liquid N_2 reservoir to improve pump-down time.
10. Confirm metal **parameters** on each layer to be used (density, tooling, z-factor, etc.).
11. Program the **thickness** for each layer in nm.

Evaporation:
1. Wait until vacuum $\leq 2x10^{-7}$ Torr is obtained.

2. Turn electron **Gun Power Supply ON**.
3. Turn **gun** and **on/off ON**, and wait for lights (Power, Vac, H_2O, Rot, Gun, Local, and Beam).
4. **Check** ~4.85 kV high voltage setting and 15-17 °C water chiller.
5. Using **Data** button, select appropriate layer and confirm settings changing as necessary.

	Ti	Al	Au	Ni
Layer	1	3	2	4
Density	4.5	2.7	19.3	8.91
Z-factor	14.1	8.2	23.2	26.6
Tooling	0.85	0.85	0.85	0.85
Beam for evap	120 mA	45 mA	100 mA	120 mA

6. Ensure shutter is **closed** and **no** shutter control buttons are pushed.
7. Activate **Beam Sweeping** by setting control knob to "1".
8. Turn **Beam Current** control knob to 1st notch (~ 20 mA).
9. Slowly ramp **Beam Current** up in 20 mA steps every several seconds, monitoring vacuum pressure—don't let pressure exceed $1x10^{-5}$ Torr.
10. If metal has not been used recently, **evaporate off impurities** by getting metal liquid hot (i.e., at the onset of evaporation—watch for solid to liquid phase change) otherwise go to step 12.
11. As necessary, allow chamber to return to $2x10^{-7}$ Torr, and then repeat starting at step 9.
12. Stop ramping **Beam Current** when desired beam current is achieved, or turn down if $9x10^{-6}$ Torr is exceeded.
13. Arm shutter by depressing **Remote** button
14. Press "**Run**" to open shutter and start evaporation, noting start time.
15. Watch deposition rate and pressure; modify **Beam Current** to keep both within proper limits.
16. **Log** time when deposition completes.
17. Turn **Beam Current** down slowly (2-3 seconds) to zero.
18. Activate **Carousel** and move to position of next metal, else go to step 21 if done evaporating.
19. Using **Data** button, select appropriate layer and confirm settings changing as necessary.
20. Go to Evaporation step 8 when chamber returns to $2x10^{-7}$ Torr
21. Deactivate **Beam Sweeping**, **Carousel**, and disarm shutter **Remote**.
22. Turn **gun** and **on/off OFF**, turn **Gun Power Supply OFF**.
23. Log evaporation results into the Evaporation log book.
24. Wait at least ½ hour and follow vent procedures.

Appendix E

Photoluminescence Procedures

Mounting and Inserting Samples:
1. Mount up to three 5 mm x 5 mm samples on each side of copper block using dot of rubber cement on top back.
2. Carefully slide rod into chamber, reconnect TC gauge wire; secure w/clamp.
3. When sample chamber is already under a vacuum:
   - **Close valve** on top Cu pipe to sample chamber.
   - **Connect** pressurized He line to small red lever and **open valve**.
   - **Undo clamp** on sample rod. When chamber is pressurized w/He, cap will pop.
   - Complete steps 1 & 2 above.
   - **Close** small red lever; **open** valve on top Cu pipe, turn **off He tank** & disconnect line.

Initial Start-up (everything at 760 Torr and room temperature):
1. **Turn on roughing** pump to rough out vacuum jacket through dormant turbo, ensuring valve to jacket is open.
2. When jacket < 100 μm Hg, **turn on turbo** pump on top shelf (after ~ x hours of roughing).
3. When jacket < 2 μm Hg, **turn on big roughing** pump at wall to rough sample chamber (~ x hrs of turbo).
4. Ensure vacuum release valve is closed (black knob by chamber gauge) and **open valve** on top Cu pipe.
5. **Purge LHe reservoir** w/pressurized He:
   - **Connect He gas** tank to inlet of LHe reservoir;
   - **Open black knob** at top of chamber & put "+" pressure into resvr;
   - **Close black knob & open valve** on bottom Cu pipe (rough pump pulls on LHe resvr & pump gurgles); **Close bottom pipe valve**.
6. Perform step 5 three times to **fully purge** LHe resvr.
7. **Open black knob** at top of chamber & put "+" pressure into resvr again.
8. **Open needle valve** to pull pressurized He through sample chamber (pump will gurgle); **close needle valve**.
9. **Close vacuum jacket valve** to turbo just before adding LN_2 to avoid cryo-pumping through turbo into jacket
10. **Dump LN_2** into LN_2 resvr maintaining "+" pressure on LHe resvr.
11. **Test needle valve** to ensure free LHe flow to chamber (pump gurgles).
12. When LN_2 resvr is full and stops bubbling over, **close black knob** to LHe resvr & **disconnect He gas** tank
13. **Wait** at least 3 hours and **top off LN_2** resvr before adding LHe to resvr.

Adding LHe: (everything under a vacuum and LN_2 reservoir full for > 3 hours)
1. Position LHe dewar and **Connect He gas** tank to dewar; turn on **LHe meter**.
2. Open top dewar valve and **insert transfer tube** to LHe level (watch dewar pressure and add as necessary).
3. When LHe begins to "puff" from transfer tube, **insert** into LHe resvr inlet; **remove rubber stop** on exhaust.
4. Dewar pressure determines transfer rate. **Test needle valve** to ensure free LHe flow to chamber (pump gurgles).

5. When meter reads appropriate LHe level, quickly **raise dewar tube** above LHe level.
6. Quickly pull LHe resvr **tube fully out** and **cap** with valve fixture.
7. **Store** transfer tube, **close** dewar valve, **disconnect** He tank; **replace** rubber stop on LHe resvr exhaust.
8. **Adjust needle valve** so that sample chamber pressure ~ 23 mm Hg to begin cooling samples, monitor T_A, T_B.

System Start-up (everything under a vacuum and LN_2 & LHe reservoirs full):
1. **Start N2 purge**: open N2 tank & set flow to ~5 on GF-1 unit.
2. Turn yellow **H_2O lever on**; turn **chiller power on**, press **green start** button.
3. Put on appropriate **safety goggles**; turn on laser **warning light**.
4. **Turn key** on argon ion laser controller, **ramp up current** to ~50 A, wait until laser is visible.
5. Set laser **output power** to appropriate wattage.
6. Ensure **LN_2 dewar is full** and pressurized (5-8 psi)
7. Turn on **PMT cooling** system; set to $-40\ ^{o}C$.
8. After PMT is cooled, **set HV** to 1700 V from within SPEX PC program.
9. Run **test spectra** to check noise level in PMT.

Collect Spectra (when laser power, sample temp, and PMT temp are at appropriate levels):
1. T = 3K spectra requires T_A ~5.7 K and T_B ~ 1.8 K (chamber pressure will be ~18-23 mm Hg)
2. **Set slits** appropriately (100-400 μm) depending on sample intensity & resolution desired.
3. **Illuminate sample** & send reflected beam into corner of sample dewar ***before*** window to spectrometer.
4. Select a spectral feature & **optimize** luminescence intensity into spectrometer by laterally adjusting 2nd lens.
5. **Run scan** using SPEX program (3.6-1.8 eV, 2 Å step, 0.05 s integration).

Variable Temperature Spectra:
1. Run lowest temp spectra first, then collect spectra as T increases.
2. Adjust needle valve to stabilize at $T < 100$ K.
3. Adjust needle valve and sample heater to stabilize at $T > 100$ K.

Variable Power Spectra:
1. Set laser for highest power spectra into sample dewar; measure w/meter; collect spectra.
2. Set up neutral density filters just before sample dewar; use UV ND filters to collect lower power spectra.

System Shut-down:
1. **Close needle valve** to LHe resvr.
2. Turn laser **power down** to zero; turn **key off**; wait 10 min & turn **chiller off** and **H_20 off**.
3. Turn N2 purge gas **tank off**.
4. Turn **off PMT HV** from within SPEX PC program.
5. Turn **off PMT cooling** system.
6. Ensure **pressure release** valve on LN_2 dewar is closed.
7. **Close shutter** to spectrometer entrance to keep dust out.
8. **Top off LN_2** resvr if you want to keep vacuum jacket at usable vacuum level.

Cathodoluminescence Procedures

1. Ensure both Spectrometer computers are turned **ON**
2. Check chamber pressure with ion gauge (nominally 1.6×10^{-6} Torr), then turn **OFF** ion gauge so filament light doesn't corrupt CL signal.

CAUTION: Should have $P \sim 10^{-6}$ Torr prior to turning on gun or cooling samples to maintain cathode life and prevent condensation on samples. Chamber can get to 10^{-7} Torr range after pumping overnight; can get to 1.2×10^{-7} Torr after weeks of pumping.

PMT and electron gun start-up:

3. Turn **ON** PMT cooler (set for -40 to –35 °C)
4. Turn **ON** Stanford high voltage for PMT (trip ON)
5. Turn **ON** Gamma box with single toggle
6. Turn **ON** Kimball unit (turn key and light 4 buttons)
7. Set Beam Energy to 5 keV
8. Close Faraday cup to monitor Beam Current
9. Slowly ramp source current UP in 0.5 A increments every several seconds until cup current reaches desired level. Note: Kimball Source Current = 2.43 A at E=10 keV, T=10 K for 50 µA.
10. Wait for spectrometer scan to stabilize at noise level (10^{-5} µA) before collecting spectra

Low-temperature set-up (do this in parallel with PMT and electron gun start-up):

11. Plug in heater to prevent icing at tip exhaust
12. Slowly lower transfer tube down into He tank ensuring pressure 5-8 psi
13. Open shield and tip flow valves two full turns (Note: T may go up beginning to drop ~ 20 min later)
14. Insert transfer tube into the He tank as necessary and adjust flow valves to maintain 10-12 K temperature (nominal flow meter readings of 3)

Shut down:

1. Pull transfer tube above liquid level in He tank. Caution: Do not break seal and lose pressure.
2. Close flow meter valves finger tight
3. Turn **OFF** PMT cooler
4. Turn **OFF** Stanford high voltage for PMT (trip **OFF**)
5. Slowly ramp source current Down to 0 A in 0.5 A increments every several seconds
6. Set Beam Energy to 0 keV
7. Turn **OFF** Kimball unit (4 buttons ending with "power" and turn key OFF)
8. Turn **OFF** Gamma box with single toggle
9. Leave spectrometer computers **ON**
10. Turn monitor **OFF**
11. Unplug heater after chamber T=100 K

Vent chamber, change samples and reload:

CAUTION: Ensure chamber is at room temperature and electron gun is fully **OFF** for at least 1 hour.

1. Toggle stop switch on 450 Turbo pump
2. If green lights on vent valve controller don't come on after 2 minutes, cycle power to vent valve controller
3. Place magnet on fitting and open valve (Turbo pump winds down, keep magnet there so N_2 can vent chamber)
4. Disconnect Temperature gauge and Tip exhaust from heater/sample holder
5. Pull cold finger out holding heater down while waiting for vacuum to break
6. Place finger on top of chamber depressing the end of transfer tube
7. Once vacuum breaks, close vent valve using magnet and return magnet to holder
8. Carefully pull sample holder straight out of chamber
9. Change out samples using sharp stick to apply rubber cement to back of samples
10. Replace sample holder carefully, lowering without pinching the two O-rings
11. Manually switch roughing pump **ON** (it will beep and chug)
12. Replace cold finger carefully into chamber top all the way and tighten a couple of threads
13. Wait for TCs to read in the low 10^{-3} Torr range, then turn roughing pump switch **OFF**
14. Turn **ON** Turbo pump at front panel (yellow light on)
15. Turn **ON** ion gauge to confirm vacuum is building ($P<10^{-3}$ Torr)
16. Turn **OFF** ion gauge and TC should read $1\text{x}10^{-3}$ Torr

Increase pressure in He tank then change when empty:

1. Change fitting on vent valve to accept small tube on standing He tank
2. Add He until pressure on main He tank reads 6-8 psi
3. Close vent valve, close standing tank valve, and remove flex tube
4. Repeat steps 2 and 3 once more when pressure and flow become too low
5. Replace standard fitting on vent valve
6. Vent main He tank when T begins rising and main tank pressure is < 1 psi,
7. Remove transfer tube
8. Close both tank valves, and label as residue
9. Clean frost off transfer tube, close vent valves, open top valve and insert transfer tube maintaining < 10 psi
10. Secure transfer tube with brass fittings and clamp at proper height

Collect spectra:

1. Ensure PMT T ~ –38 to –40 °C and spectrometer noise level has stabilized in 10^{-5} μA range
2. Ensure sample T is stable at desired measurement T
3. Ensure Beam Energy, all slits, and Beam Current are at desired levels at measurement T
4. Position E beam on sample center using X & Y controls; adjust Focus as necessary to control beam size/shape
5. If not already known, set scan parameters (F4) and run a quick spectrum (20 Å steps) to identify largest peaks
6. Set spectrometer to look at the most intense energy/wavelength (F9)
7. Optimize signal at the peak E/λ by adjusting lenses and X & Y controls; record max luminescence current
8. Set nominal scan parameters (3.6—1.8 eV at 2 Å step for GaN samples) and collect data
9. Output all *.spt files to *.txt ASCII files and copy to A:\

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13. SUPPLEMENTARY NOTES

14. ABSTRACT

Recently, research on the wide bandgap semiconductors such as GaN and $Al_xGa_{1-x}N$ became very popular for their applications on various devices. Therefore comprehensive and systematic luminescence studies of Si implanted $Al_xGa_{1-x}N$, Mg doped GaN, and Si+N implanted GaN grown on sapphire substrates by molecular beam epitaxial method have been made as a function of ion dose and anneal temperature. The ions were implanted at 200 keV with doses ranging from $1x10^{13}$ to $1x10^{15}$ cm^{-2} at room temperature. The samples were proximity cap annealed from 1200 to 1350 °C with a 500 Å thick AlN cap in a nitrogen environment. It has been found that the optical activation and implantation damage recovery are highly dependent upon ion dose and anneal temperature. The results of luminescence measurements on $Al_xGa_{1-x}N$ made at 5 K by both photoluminescence and cathodoluminescence show that nearly complete implantation damage recovery can only be obtained after annealing at 1350 °C. The Si+N implanted GaN showed only a small amount of optical activation in the cathodoluminescence measurements at 1250 to 1350 °C. Mg doped GaN results indicated that optical activation of the Mg ions was not prevalent, and ion implantation damage might not have been removed completely at 1350 °C. Electrical measurements indicated that the p-type GaN changed to n-type after annealing, so no further measurements were taken for the Mg-doped GaN. The results also indicate that current AlN cap protected the implanted $Al_xGa_{1-x}N$ layer very well during high temperature annealing without creating any significant annealing damage. These luminescence observations are consistent with the results of electrical activation studies made on these samples.

15. SUBJECT TERMS

Luminescence, GaN, AlGaN

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